

RCRA LABORATORY COMPLIANCE EVALUATION INSPECTION CHECKLIST

**DETREX CHEMICAL INDUSTRIES, INC.
DETROIT, MICHIGAN
MID 091 605 972**

Prepared for:

**U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Waste Programs Enforcement
Washington DC 20460**

Prepared by:

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EPA Region	:	5
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ABBREVIATIONS AND ACRONYMS

CF	Correction factors	KI	Potassium iodide
Cl	Chloride	LAI	Laboratory audit inspection
CLP	<i>Contract Laboratory Program</i>	M	Molar
cm	Centimeter	MDL	Method detection limit
CN ⁻	Inorganic Cyanide	MDNR	Michigan Department of Natural Resources
CWA	Clean Water Act	μg	Microgram
°C	Degrees Celsius	μL	Microliter
ECD	Electrolytic conductivity detector	μm	Micron
EP	Extraction procedure	mg/kg/min	Milligram per kilogram per minute
EPA	Environmental Protection Agency	mg/L	Milligram per liter
EPTOX	Extraction procedure toxicity	mL	Milliliter
eq	Equivalent	mL/g	Milliliter per gram
FID	Flame ionization detector	ml/min	Milliliter per minute
g	Gram	N	Normal
GC/MS	Gas chromatograph/mass spectrometer	NA	Not applicable
H ₂ O	Water	NaOH	Sodium hydroxide
H ₂ S	Hydrogen sulfide	NIST	National Institute of Standards and Technology
H ₂ SO ₄	Sulfuric acid	nm	Nanometer
HCl	Hydrochloric acid	PID	Photoionization detector
HCN	Hydrogen cyanide	psi	Per square inch
HNO ₃	Nitric acid	QA	Quality Assurance
ID	Identification	QC	Quality Control
KCN	Potassium cyanide	RCRA	Resource Conservation and Recovery Act of 1976
Kg	Kilogram	RF	Response factors
		rpm	Revolutions per minute
		RSD	Relative standard deviation

ABBREVIATIONS AND ACRONYMS (Continued)

SEC	Seconds
SOP	Standard operating procedure
TCLP	Toxicity characteristic leaching procedure
ZHE	Zero headspace extraction

SECTION I GENERAL INFORMATION

Facility Name: Detrex Chemical Industries, Inc.
Facility ID No.: MID 091 605 972
Reviewer: C. Friday / N. McDonald
Date Completed: 5-18-95

1.0 CONTACTS

EPA Facility ID No.: MID 091 605 972

EPA Region: 5

Facility Name: Detrex Chemical Industries, Inc.

EPA Contact: Sue Rodenbeck Brauer

Telephone No.: (312) 353-6134

Address: 12886 Eaton Avenue
Detroit, Michigan 48227

MDNR Contact: Jeanette Nouchel

Telephone No.: (313) 953-1408

Laboratory Name: Detrex Chemical Industries, Inc.

Laboratory Contact: Andres Garcia

Phone Number: (313) 491-4550

Address of Laboratory: 12886 Eaton Avenue
Detroit, Michigan 48227
(313) 491-4550

Name, address, phone number, and project manager of auditing firm:

PRC Environmental Management, Inc.

1035 Dairy Ashford Rd. Suite 240

Houston, Texas 77079

Candace D. Friday, 713/589-6477

Names of inspectors: Nancy McDonald - PRC

Candace Friday - PRC

Dates of inspection: 5-17-95

2.0 FACILITY STATUS (To be completed jointly with MDNR and U.S. EPA prior to inspection)

Does the owner/operator have the following:

Interim Status (Go to Section 2.1)

- ☐ Detection monitoring
- ☐ Assessment monitoring
- ☐ Corrective action (Section 3008h)

Permit Status (Go to Section 2.3)

- ☐ Detection monitoring
- ☒ Compliance monitoring
- ☒ Corrective action

2.1 Past Action Taken at Facility (Interim Status)

Type

- Operation and maintenance
- Comprehensive groundwater monitoring evaluation
- Case development inspection
- RCRA facility assessment
- Compliance evaluation inspection
- Reviews of the laboratory (such as CWA, LAI, or CLP)
- Performance audit samples

Dates

Not applicable

Complete the following regarding the actions listed above:

- Does MDNR have copies of completed inspection reports and site studies?

☐ Yes

☐ No

- For each action listed above, summarize any deficiencies regarding the owner/operator's sampling and analysis program, specifically, any deficiencies relating to the owner/operator's ability to generate high quality monitoring data.

Go To Section 2.2

2.2 Identify enforcement actions issued to the facility regarding to interim status violations

Action

- Section 3008(a) complaint/order
- Section 3013 order
- Section 3008(h) complaint/order
- Section 7003 complaint/order
- Referral for litigation

Dates

Not applicable

For each action listed above, state whether the enforcement action focused on the owner/operator's sampling and analysis program. Summarize relevant requirements imposed on the owner/operator.

Go To Section II

2.3 Actions Taken at the Facility to Date (Permit Status)

Type

Dates

- Permit issuance
- Operation and maintenance inspection
- Comprehensive groundwater monitoring evaluation
- Case development inspection
- Compliance evaluation inspection
- RCRA laboratory audit inspection

June 30, 1992

3 times per year

1 time per year

Complete the following regarding the actions listed above:

- Does MDNR have a copy of the permit and copies of inspection reports completed after permit issuance?

☒ Yes

☐ No

- Summarize deficiencies identified after permit issuance regarding the owner/operator's sampling and analysis program

Manifest MI3351113 - did not conduct fingerprinting for 13 days after waste received - waste was analyzed off-site (Euclid, OH, Detriex facility). Later determined no compatibility testing was performed on sample (performed at Euclid OH)
Failure to reprofile generator on a yearly basis.

Go To Section 2.4

2.4 Identify enforcement actions issued to the facility after the permit issuance date

Action

- Section 3008(a) complaint/order
- Section 3008(h) complaint/order
- Section 3013 order
- Section 7003 complaint/order
- Referral for litigation
- Permit revocation

Dates

Not applicable

For each action listed above, state whether the enforcement action focused on the owner/operator's sampling and analysis program. Summarize relevant requirements imposed on the owner/operator.

Go To Section II

SECTION II SAMPLING AND ANALYSIS PLAN

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday, N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Determine if the owner/operator's sampling and analysis plan includes the following:				
Matches the most current plan used at the site and provides the date of the plan?	✓			
Names an individual as the laboratory quality assurance manager and specifies job requirements for the position?	✓			
Includes a current summary of training, experience, and job description required for each member of the laboratory staff?	✓			
Describes quality control paperwork flow and identifies those who are authorized to approve data and results?	✓			
Identifies personnel responsible for corrective action procedures?	✓			
Describes the laboratory's system for developing or revising technical procedures and identifies those who have authorization to do so?	✓			
Requires dating chemicals upon receipt and using them on a first-in, first-out basis?		II-1		II-1 These items are not included in the waste analysis plan.
Specifies use of reagent-grade or high-purity chemicals to prepare standards?				
Requires testing of chemicals used in analyses to ensure they contain no contaminants that may interfere with analyses?				
Requires labeling of all reagents and solutions to indicate identity, concentration, storage requirements, preparer's name, preparation date, and expiration date?				
Requires routine checking and recording of the conductivity of distilled and demineralized water?				
Specifies use of reagent-grade water, as required by the specific method?				
Specifies use of distilled water, as required by the specific method?				
Requires discontinuing the use of any reagents or solutions labeled with expiration dates that have passed?			✓	

SECTION II SAMPLING AND ANALYSIS PLAN (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Requires storage of samples and standards containing analytes of interest in areas other than those where trace analysis is performed?		II-1		<p>laboratory Quality Assurance</p> <p>II-2 The waste analysis plan specifies QC samples will be analyzed at a frequency of 1% or one per day, whichever is more frequent.</p>
Requires storage of standards separately from sample extracts?		II-1		
Specifies the use of analysis request sheets or work orders?	✓			
Includes and requires the use of written calibration procedures, analytical procedures, computational procedures, quality control procedures, and operating procedures?	✓			
Requires daily instrument calibration?	✓			
Specifies the use of standard curves and check samples for calibration purposes?	✓			
Specifies the use of logs to record all instrument and equipment checks?		II-1		
Describes when an analytical system is "out of control" through internal quality control samples?	✓			
Requires corrective procedures when an analytical system is "out of control"?	✓			
Specifies the use of Class A glassware?		II-1		
Names a sample custodian in the laboratory?	✓			
Describes storage requirements for incoming samples?	✓			
Specifies the assignment of unique laboratory numbers to all incoming samples?	✓			
Requires maintenance of proper temperatures for incoming samples?	✓			
Describes chain-of-custody procedures that the laboratory will use?	✓			
Specifies the use of a master schedule sheet or logbook of all samples being analyzed, indexed by laboratory numbers, client, date of arrival, and analysis to be performed?	✓			
Specifies maximum holding times for samples?	✓			
Requires the daily temperature recordings in cold storage areas?		II-1		
Specifies the use of matrix spikes (one per analytical batch per matrix, or one per every 20 samples, whichever is more frequent)?		II-2		

SECTION II SAMPLING AND ANALYSIS PLAN (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Requires the use of laboratory duplicates (one per analytical batch per matrix, or one per every 20 samples, whichever is more frequent)?		II-2		<p>II-3 A single chemist acts as lab manager, QA officer, lab staff, and sample custodian. Therefore, there is no independent review of raw data</p> <p>II-4 Data from independent labs are not validated by Detref.</p>
Requires the use of blanks (one per analytical batch per matrix, or one per every 20 samples, whichever is more frequent)?				
Requires the use of field duplicates (one per analytical batch, or one per every 20 samples, whichever is more frequent)?				
Requires the use of check samples (one per analytical batch, or one per every 20 samples, whichever is more frequent)?				
Requires the use of surrogates for volatile and semivolatile organics and pesticides (added to every blank, standard, sample, and quality control sample)?		✓		
Requires the use of column check samples (absorbent chromatography and back-extractions of organic compounds with one per batch of absorbent)?			✓	
Requires the use of standard curves for analytical methods?	✓			
Requires gas chromatograph/mass spectrometer instrument performance check (in which the initial five-point calibration is verified with a single-point calibration once every 12 hours of instrument operation and, if the sensitivity and linearity criteria are not met, a new five-point initial calibration must be generated)?			✓	
Requires owner/operator to have a system that independently examines and validates raw data from the laboratory?		II-3		
Requires owner/operator to have a system that examines and validates raw data when a commercial laboratory is used?		II-4		

SECTION III PERSONNEL

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Review the laboratory's personnel qualifications and organizational structure.				<p>III-1 A single chemist functions as lab manager, QA officer, lab staff, and sample custodian.</p>
Has the laboratory appointed a quality assurance manager who routinely performs the following actions:	III-1			
- Ensures adherence to quality assurance requirements for sampling?	✓			
- Ensures that all test and measuring equipment are properly calibrated?	✓			
- Monitors logging in of samples?	✓			
- Approves project plans, specific analyses, and final reports?	✓			
- Maintains a copy of the master schedule sheet?	✓			
- Maintains separate copies of all methods performed by the laboratory?	✓			
- Maintains written and signed records of periodic inspections?	✓			
- Maintains all quality assurance records in one location?	✓			
Are qualified individuals used to perform the required analyses?	✓			
Are qualified individuals authorized to approve data and results?	✓			

SECTION IV PHYSICAL INSPECTION

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Review relevant documents as detailed below, and if possible, observe the use of these documents during receipt of a shipment.				
Is a copy of the SOP for receipt of shipments available to the receiver?	✓			
Is the SOP used consistently?				
Are approved waste characterization reports or similar documents used by receiver to identify the particular waste?				
Are the necessary tools available to open containers to check the waste?				
Are hazardous waste manifests and other shipping documents used as the starting point for the receiving procedures?				
Is the manifest immediately reviewed to determine whether the shipment is at its proper destination?				
Is the shipment immediately compared to the manifest to determine if there are any discrepancies?				
Is there an immediate attempt to resolve discrepancies?				
Is the manifest signed and dated, noting unresolved discrepancies, with one copy given to the transporter?				
Are the other copies of the signed manifest filed internally for proper disposition?				
Is the shipment identified against an approved waste characterization report or similar document?				
Is the entire shipment or portions of the shipment inspected?				
Are the color and consistency of the waste checked and recorded?				
During shipment inspection, are unusual odors of the waste recorded?	✓			

SECTION IV PHYSICAL INSPECTION (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are vapor screening techniques used to evaluate vapors?			✓	IV-1 The facility is just beginning to set up a computerized system for information management. Presently, a master logbook is being used to track incoming shipments.
Are the shipment and its description logged into the facility's information management system?		IV-1		
Are procedures in place to verify a shipment that does not meet the standard description?	✓			
Are procedures in place to verify the proper sampling and testing of the waste?	✓			

SECTION V SAMPLING

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Euday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
1.0 DOCUMENTS Review relevant documents, as detailed below, and if possible, observe the use of these documents during sampling.				
Does the facility's waste analysis plan specify which analyses will be performed on which incoming wastes?	✓			
Does a written SOP (or similar document) translate these requirements into specific methods for sampling?	✓			
Does the written SOP identify required apparatus, reagents, and procedures?	✓			
Is the SOP available for use by the sampler?	✓			
Is the SOP used?	✓			
2.0 APPARATUS AND MATERIALS				
Does the SOP specify sampling equipment for all types of incoming shipments?	✓			
Is the equipment available?	✓			
Is the equipment properly decontaminated between shipments?	✓			
Does the SOP specify proper sample containers and preservatives?	✓			
Are the sample containers available and cleaned according to EPA protocol?	✓			
Does the SOP specify labels?	✓			
Are the labels complete (including waste type, waste lot number, sampler, sampling time and date, analyses to be performed, and, preservatives, if any)?	✓			
Is adequate secured storage space available at the appropriate temperature for the samples awaiting analysis?	✓			
Are sample preservatives used?			✓	
If so, are they readily available?			✓	

SECTION V SAMPLING (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 PROCEDURE - BULK SHIPMENTS				
Are composite samples collected?			✓	
Is the sampling scheme random, systematic random, systematic, or not specified? (Explain in Comments)			✓	
4.0 PROCEDURE - CONTAINERIZED SHIPMENTS				
Does the facility specify how many containers in a shipment will be sampled?	✓			
Are the containers selected randomly?			V-1	
Are samples from multiple containers in a shipment composited?	✓			
Is the sample from a container collected by a method that is random, systematic random, systematic, or not specified? (Explain in Comments)			✓	
5.0 SAMPLING				
Are adequate tools available to get to the sampling point (including container handling equipment, barrel wrenches, and pipeline taps)?	✓			
Are adequate tools and equipment available for sampling (including coliwesas, triers or thieves, sampling bombs, and ladles)?	✓			
Are samples properly collected and composited?	✓			
Are sample containers filled and labeled as specified?	✓			
Are samples properly logged into the facility's information management system and securely stored until analysis?		IV-1		
Is the shipment properly stored until analyses have been completed and an informed decision has been made?		V-2		
If a shipment appears to be variable, are separate samples prepared and analyzed?	✓			

V-1 All drums are sampled; up to 10 samples may be composited.

V-2 Drums were unloaded before the fingerprint analysis was performed and the manifest signed. This is a permit violation.

SECTION VI GENERAL LABORATORY SUPPLIES AND EQUIPMENT

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Review the procedures for maintaining the laboratory's supplies and equipment.				
Are adequate laboratory facilities and instruments available to perform the required analyses?	✓			<p>VI-1 On the day of the audit, these items were not addressed because the only lab personnel was not available.</p> <p>On January 23, 1996 Detrex supplied the response to these items. The new results are indicated with an asterisk (*).</p> <p>VI-2 The response indicated that the distilled water was purchased and certified by the supplier. They do not use any grade of water for organic analyses.</p>
Is the solvent storage area properly vented and appropriate for the prevention of possible laboratory contamination?	✓			
Are analytical and sample storage areas isolated from all atmospheric sources of solvent?	✓			
Are chemicals dated upon receipt and used on a first-in, first-out basis?	✓			
Are reagent-grade or high-purity chemicals used to prepare standards?	✓			
Are chemicals used in analyses tested to ensure that they contain no contaminants that may interfere with the analyses?	✓			
Are all reagents and solutions labeled to indicate identity, concentration, storage requirements, preparer's name, preparation date, and expiration date?	✓			
Is a source of distilled or demineralized water available?	✓*	VI-1		
Is the conductivity of distilled or demineralized water routinely checked and recorded?			VI-2*	
Is reagent-grade water used for organic methods?			VI-2*	
Is distilled water used for inorganic methods?	✓*	✓		
Are any of the reagents or solutions being used labeled with an expiration date that has passed?		✓		
To avoid contamination, are samples and standards containing the analytes of interest stored or used in areas other than those where trace analysis is performed?	✓			
Are standards stored separately from sample extracts?	✓			

SECTION VI GENERAL LABORATORY SUPPLIES AND EQUIPMENT (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Do chemical handling areas consist of either a stainless-steel bench or an impervious material covered with absorbent materials?	✓			
Are contamination-free areas provided for trace level or organic analytical work?	✓			
Are exhaust hoods provided to allow contamination-free work with volatile materials (that is, venting for preparation, extraction, and analysis)?	✓			
Is an adequate supply of routinely needed in-house replacement parts available to ensure that analytical equipment is not inoperable during a critical period?	✓			
Is a service record logbook maintained for each analytical instrument?	✓			
Are instruments properly vented and appropriate traps in place, as required?			✓	
Are chemical waste disposal policies and procedures well-defined and followed by the laboratory?	✓			
Is Class A glassware used or is it calibrated to ensure that the amount marked on the glassware coincides with the amount delivered?	✓			
Is the glassware checked periodically to ensure that calibration is correct?	✓			
Is the glassware cleaned correctly after each use to ensure that there will be no contamination with the next use?	✓			
Is the analytical balance located away from drafty areas and areas subject to rapid temperature changes?	✓			
Has a certified technician calibrated and checked the balance within 1 year?	✓			

SECTION VII SAMPLE RECEIPT AND STORAGE

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: _____

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Review sample handling procedures at the laboratory.				
Is a sample custodian appointed to log incoming samples?	✓			
Is a written SOP available that describes sampling requirements (such as, type of sampling container, preservation technique, and storage container) for each analysis?	✓			
If no custodian is appointed, are the individuals logging in samples aware of the sampling requirements for each analysis?			✓	
Does the custodian know the process for storing incoming samples?	✓			
Is a sample label affixed to each container?	✓			
Do sample labels contain information sufficient to identify the sample and ensure that it has been sampled in the correct manner (including facility name, station number, date sampled, time sampled, type of analysis requested, preservation used, and signature of sampler)?	✓			
Are samples collected in the type of container specified for each analysis?	✓			
Are samples preserved as required and cooled to 4°C?	✓			
Do samples shipped to the laboratory arrive at the correct temperature to ensure that the sample has remained in a preserved state?			✓	
Are water samples for volatile analyses checked for air bubbles?			✓	
Are trip blanks, field blanks, and field duplicates used as required?			✓	
If so, are they identified as such?			✓	
If used, are spiked samples identified?			✓	
Is a chain-of-custody form filled out and kept on file?	✓		✓	
Is the information on the sample tag and chain-of-custody form verified and matched?	✓			
Are unique laboratory numbers assigned to all incoming samples (including quality control samples)?	✓			

SECTION VII SAMPLE RECEIPT AND STORAGE (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Does the laboratory maintain a master schedule sheet or logbook of all samples being analyzed, indexed by laboratory number, client, date of arrival, and analysis to be performed?	✓			<p>VII-1 Because the lab is very small, separate work orders are not necessary.</p> <p>VII-2 Temperature records were not available; however, samples are analyzed very soon after collection so that storage temperature is not critical.</p>
Is the laboratory number written on the sample label, the master schedule sheet, and any documents related to that sample?	✓			
Are completed sample analysis work orders available for each sample?	✓			
Does each sample have a separate work order for each analysis or group of analyses (that is, organic and inorganic) to be performed (to ensure that each analyst who must perform an analysis on that sample will have a work order)?		VII-1		
After all analyses have been completed, are all work orders attached to all appropriate summary sheets for each analyses?			✓	
Are all samples analyzed within required holding times?	✓			
Are samples maintained at the correct temperature until the time of analysis?	✓			
Are adequate facilities provided for storage of incoming samples, including cold storage?	✓			
Are volatile samples stored separately from nonvolatile or semivolatile samples?			✓	
Is the temperature of the cold storage recorded daily in a logbook?		VII-2		
Are temperature outside of control limits noted, and are appropriate actions taken when required?				
If reused, are sample containers cleaned properly?			✓	
Are the possession and handling of samples traceable from the time and date of collection to the time and date of analysis and reporting?	✓			
Demonstrate by tracing three samples available in the laboratory. Summarize by completing Form VII-1 (see page VII-3).				

SECTION VII SAMPLE RECEIPT AND STORAGE (continued)

FIGURE VII-1
Sampling Tracing Form

Use this form to demonstrate the traceability of samples from sample collection to reporting. Sample A should be from the oldest waste stream and should be traceable from inception of waste handling; Samples B and C should be samples from most recent 3 years. If an external laboratory is used, trace one sample to the external laboratory and back, and review the external laboratory data.				
Sample ID Information	Sample A	Sample B	Sample C	Comments (Explain all negative responses)
Sample Number	3472008	Wastewater	001a	The operations manager stated that data from Clayton Labs delivered in electronic format was did not match raw hardcopy data. Subsequently, Detroit implemented a system to check electronic data against the raw data.
Facility Name	AAR Cadillac	Detroit	Detroit	
Facility Address	Cadillac, MI	Detroit, MI	Detroit, MI	
Sample Location	drum	Wastewater grab	Station # 2 air sample	
Sampler Name	# 3		Ron Hritzglowin	
Date Sampled	3-30-95	5-1-95	3-28-95	
Time Sampled	—	—		
Recipient at Laboratory (Indicate if external)	In-house	5-2-95	Clayton Labs	
Date Received	3-30-95	RTI Lab	4-4-95	
Laboratory Number	10625 GR	95-1447A	001a 002a	
Analyses Requested	GC, compatibility, Rotemp, & ignita.	pH, CN, FOG, TSS, BOD, metals		
Storage Procedures	—	—	—	
Date of Analysis	3-30-95	5-9-95	4-17-95	
Analysts ID	AG	Signed	ES	
Methods Used	SOPs in house	150.1, 335.8, 413.1, 200.7	Niosh	
Date Results Reported	3-31-95	5-9-95	4-17-95	

SECTION VIII QUALITY CONTROL PROGRAM

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Fuday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
1.0 GENERAL QUALITY CONTROL				
Indicate the estimated daily sample throughput for the laboratory.				→ 10-20 samples per day.
Is one matrix spike used for every analytical batch or every 20 samples, whichever is most frequent?		II-2		
Are matrix spike accuracies analyzed to establish that the analytical measurement system is functioning properly with the desired sensitivity?	✓			
Are precision results of sample replicates measured for each method to indicate reproducibility among individual measurements of the same property under similar conditions?	✓			
Are the precision and accuracy results used to determine the control limits for all operating parameters?	✓			
Are these precision and accuracy results organized in the form of quality control charts?		VIII-1		
Are quality control charts or tabulation of mean and standard deviation (or the equivalent) used to document the validity of data on an as-run basis?		↓		
Are matrix spike results compared to control charts on an as-run basis to determine whether the analysis is "in control"?		↓		
Is one check sample used per analytical batch or every 20 samples, whichever is more frequent?		II-2		
Is one laboratory method blank used per analytical batch or every 20 samples, whichever is more frequent, to ensure that there are no contaminants that may interfere with the analysis?		↓		
Is one field duplicate used per analytical batch or every 20 samples, whichever is more frequent?				
Are laboratory duplicates prepared and analyzed per analytical batch or every 20 samples, whichever is more frequent (not including reinjection or reanalysis of same set of standards or samples)?		↓		

VIII-1 Control charts are not used by the lab. The lab uses established control limits from methods.

SECTION VIII QUALITY CONTROL PROGRAM (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
2.0 ORGANIC QUALITY CONTROL				
Is the analytical system calibrated each day in accordance with the requirements of the method?	✓			
Are the calibration standards analyzed and compared to control charts on an as-run basis to determine whether the run is "in control"?	✓			
Is a surrogate spike added to every blank, standard, sample, and quality assurance sample?		VIII-2		
Are column checks sample blanks used for each batch of absorbent?			✓	
Are field blanks, transport blanks, and laboratory blanks used, as needed, to ensure that the water contains no contaminants that may interfere with analysis?	✓			
Are laboratory method blanks extracted and analyzed with the same procedures used to extract and analyze samples?				
3.0 INORGANIC QUALITY CONTROL				
Is the precision of the system demonstrated by the analysis of replicate laboratory control standards each time the analytical system undergoes a major modification or prolonged period of inactivity?			VIII-3	
To prepare a standard calibration curve, are a minimum of three calibration standards covering the concentration range of the samples analyzed?				
Of these standards analyzed, is at least one at or below the required quantitation limit?				
For each day an analysis is performed, is the standard calibration curve verified by using at least one laboratory method blank and one standard curve?				
Is the daily check within plus or minus 10 percent of the original curve?				
Are laboratory method blanks used as required?				
Is the standard calibration curve verified by running one additional standard, within the range of the standard curve, every 20 samples?				
Is this check within 10 percent of the original curve?				

VIII-2 No surrogates are used.

VIII-3 Inorganic analyses are not routinely performed by the facility lab.

SECTION IX DATA HANDLING AND REPORTING

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Review procedures for data handling, reporting, and recordkeeping.				<p>IX-1 Because there is only one person working in the lab, no secondary review is performed.</p>
Are computerized and manual checks applied at various appropriate levels of the measurement process to ensure data validation?	✓			
Are the data validation criteria documented (including limits on operational parameters, calibration data, special checks, statistical tests, and manual checks)?	✓			
Does the laboratory have procedures for data handling and reporting, including the recording of data on standard forms and in laboratory notebooks?	✓			
If so, is this reporting format described with example forms provided?	✓			
Are sample calculations available for inspection?	✓			
Are bound notebooks used for all laboratory activities?	✓			
Do notebooks, logbooks, and runlogs have the following pertinent data:				
<ul style="list-style-type: none"> ✓ - Title - describing the activity being recorded ✓ - Instrumentation - if appropriate, give type and ID number (for example GC #3) ✓ - Date of preparation or analysis ✓ - Initials of preparer or analyst ✓ - For preparation notebooks or logbooks - details of activity, such as sample measurements, reagents and quantities, and procedure times, if applicable ✓ - For instruments runlogs - run sequences, identity of each sample and analyte ✓ - Units of measurements ✓ - Calculations, if applicable X - Peer or supervisory review signature and date X 		IX-1		
Are notebooks reviewed by a peer or supervisor (as indicated by a signature and date)?		IX-1		
Are raw data archived and documented properly?	✓			
Are records readily available for review?	✓			
Are records maintained for at least 3 years?	✓			

SECTION X COMPATIBILITY TEST

METHOD - Detrex SOP

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This test should evaluate wastes for compatibility with process conditions, other wastes, and construction materials.				
USAGE: Circle the sample types for which this method is used. <input checked="" type="radio"/> In-bound waste <input type="radio"/> Process control <input type="radio"/> Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for the compatibility test?	✓			
Is the SOP available at the analyst's work station?	✓			
Are bound notebooks used to record pertinent data?		X-1		
Are notebooks signed and dated by analysts?			✓	
Are notebooks reviewed and signed by a supervisor?			✓	
Are copies of notebook pages included in the final data package?			✓	
2.0 APPARATUS AND MATERIALS. Is the following apparatus available and in good working order?				
Sampling container	✓			
3.0 PROCEDURE				
Is a representative sample aliquot placed in the sampling container?	✓			
Is an aliquot of storage tank contents added to the sampling container?	✓			
Are reactions observed for 2 minutes and noted (for example, generation of heat, vapors, bubbles, or precipitate, or a change of color)?	✓			
If the sample reacts, is the shipment rejected?	✓			
4.0 QUALITY CONTROL				
If a sample result differs from the expected results, does the facility resample and reanalyze?	✓			
Does the laboratory perform duplicate analyses on selected samples?		X-2*		

X-1 Results are entered onto a summary form and filed with the manifest records.

X-2 Duplicate analyses are not analyzed routinely. They may be analyzed as a corrective action.

SECTION IX DATA HANDLING AND REPORTING

Facility Name: 'Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: _____
 Date Completed: _____

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Review procedures for data handling, reporting, and recordkeeping.				<div style="position: relative; height: 400px;"> <div style="position: absolute; top: 20%; left: 40%; transform: rotate(-45deg); font-family: cursive; font-size: 2em;"> duplicate page sm </div> </div>
Are computerized and manual checks applied at various appropriate levels of the measurement process to ensure data validation?				
Are the data validation criteria documented (including limits on operational parameters, calibration data, special checks, statistical tests, and manual checks)?				
Does the laboratory have procedures for data handling and reporting, including the recording of data on standard forms and in laboratory notebooks?				
If so, is this reporting format described with example forms provided?				
Are sample calculations available for inspection?				
Are bound notebooks used for all laboratory activities?				
Are notebooks reviewed by a peer or supervisor (as indicated by a signature and date)?				
Are raw data archived and documented properly?				
Are records readily available for review?				
Are records maintained for at least 3 years?				

SECTION XI CORROSIVITY/pH

METHOD 9040 - pH Electrometric Measurement for Liquid Samples

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: These methods are used to determine the characteristic of corrosivity (pH less than or equal to 2, or greater than or equal to 12.5). It may also be used to determine compatibility when a waste is mixed with aqueous reagents used in treatment or disposal.				
USAGE: Circle the sample types for which this method is used. <input checked="" type="radio"/> In-bound waste <input type="radio"/> Process control <input type="radio"/> Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for pH determination?		XI-1		
Is the SOP available at the analyst's work station?			✓	
Are bound notebooks used to record pertinent data?		XI-1		
Are notebooks signed and dated by analysts?			✓	
Are notebooks reviewed and signed by a supervisor?			✓	
Are copies of notebook pages included in the final data package?			✓	
2.0 APPARATUS AND MATERIALS Are the following apparatus and materials available and in good working order?				
Glass-electrode pH meter with the following? - Automatic temperature compensation - Glass-electrode plus reference electrode - Combination electrode - Low-sodium-error electrode		XI-2 XI-3*		
Beakers - 50 ml	✓			
Magnetic stirring bar and motor			✓	
3.0 REAGENTS All items must be reagent-grade or better.				
Are the following reagents available?				
- Ion-free water for cleaning electrodes			✓	
- Standard pH buffers	✓*		✓	

XI-1 No SOP for pH was available during the audit.

XI-2 During the audit, a pH meter was found in a storage area. Because the chemist was unavailable, a formal request was made to provide information regarding pH analysis.

XI-3 In response to the request, Detrex stated that the pH meter was inoperable and currently uses indicator paper. As such, reagents + procedures listed in the checklist are not applicable.

SECTION XI CORROSIVITY/pH (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are standard buffers prepared from NIST salts or <u>purchased commercially</u> (circle one)?	✓*		✓	
Do standard buffers include at least two buffers that are 3 or more pH units apart?	✓*		✓	
4.0 PROCEDURE				
Is the pH meter calibrated as instructed by the manufacturer?			✓	
Does the calibration include at least two standard buffers, that bracket the expected range and are at least 3 pH units apart?				
Is the apparatus properly set up for measurements with the following: - Electrodes fully submerged - Adequate clearance for stirring bar with no cavitation - Drift-free readings				
Are temperatures of buffer solutions determined?				
If temperatures differ by more than 2°C, is compensation applied?				
Are electrodes rinsed and wiped after each use?				
If wastes are oily or contain particulate matter, are electrodes thoroughly cleaned between samples?				
Are sample measurements repeated on different aliquots until they differ by less than 0.1 pH unit?				
Is the calibration checked at the end of a series of sample analyses?				
Is the pH meter stored with electrodes wetted?				
5.0 QUALITY CONTROL			✓	
If a sample result differs from expected results, does the laboratory resample and reanalyze?	✓			
Does the laboratory perform duplicate analyses on selected samples?		X-2*		
Does the laboratory have procedures in place to reevaluate the acceptability of a waste that does not agree with its presumed properties?	✓			

SECTION XII REACTIVITY

SW-846 Section 7.3 - REACTIVE CYANIDE AND SULFIDE PREPARATION

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method is used to determine reactivity by measuring the amount of hydrocyanic acid and hydrogen sulfide evolved upon contact with an aqueous acid. The evolved gases are collected in the scrubber solution and analyzed for cyanide by Method 9010 and for sulfide by Method 9030. See Methods 9010 and 9030 checklists for the analyses.			✓	The facility lab does not perform this method.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for reactivity preparation?				
Is the SOP available at the analyst's work station?				
Are bound notebooks used to record pertinent data?				
Are notebooks signed and dated by analysts?				
Are notebooks reviewed and signed by a supervisor?				
Are copies of notebook pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available for both cyanide and sulfide preparation:				
Round-bottom flask with three necks and ground glass joints				
Gas scrubber - 50-ml calibrated scrubber				
Stirring apparatus capable of generating 30 rpm				
Funnel with pressure-equalizing tube				
Flexible tubing				
Water- or oil-pumped nitrogen gas				
Rotometer for monitoring nitrogen gas flow rate				
Analytical balance capable of weighing to 0.001 g			✓	

SECTION XII REACTIVITY (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better.			✓	
Are the following reagents available for the evolution of hydrogen cyanide?			↓	
Reagent water - interferant-free water				
Sulfuric acid (H ₂ SO ₄) - 0.01 N				
Cyanide reference solution - 1000 mg/L				
Sodium hydroxide solution (NaOH) - 1.25 N				
Sodium hydroxide solution (NaOH) - 0.25 N				
Silver nitrate solution - 0.0192 N				
Are the following reagents available for the evolution of hydrogen sulfide?				
Reagent water - interferant-free water				
Sulfuric acid (H ₂ SO ₄) - 0.01 N				
Sulfide reference solution - 570 mg/L hydrogen sulfide				
Sodium hydroxide solution (NaOH) - 1.25 N				
Sodium hydroxide solution (NaOH) - 0.25 N				
Have reagents been analyzed for impurities?				
4.0 PROCEDURE The procedure is identical for both the evolution of hydrogen cyanide (HCN) and hydrogen sulfide (H ₂ S), except the final scrubber solution is analyzed for HCN by Method 9010 and for H ₂ S by Method 9030.				
Are 50 ml of 0.25N NaOH added to the calibrated scrubber with reagent water to fill the scrubber bottle?				
Is the system closed?				
Is the flow of nitrogen started and maintained at 60 ml/min?				
Are 10 g of waste added to the flask?				
Is the H ₂ SO ₄ solution added to the flask to bring the level to half full?				
Is the 30-minute clock started upon adding the H ₂ SO ₄ solution?			↓	

SECTION XII REACTIVITY (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are the flask contents stirred for the duration of the test?			✓	
Is the nitrogen flow stopped after 30 minutes?			✓	
Is the scrubber liquor collected and analyzed for cyanide by Method 9010 and sulfide by Method 9030?			✓	
Is amount of releasable HCN and H ₂ S and rate of release for each calculated by the following equations? $R = \text{Rate of release (mg/kg/sec)} = \frac{X * L}{W * S}$ $\text{Total releasable HCN or H}_2\text{S (mg/kg)} = R * S$ <p>where:</p> <p>X = Concentration of HCN or H₂S in scrubber solution (mg/L) from Methods 9010 and 9030, respectively</p> <p>L = Volume of solution in scrubber (L)</p> <p>W = Weight of waste used (kg)</p> <p>S = Elapsed time of reaction (sec); S = time N₂ stopped minus time N₂ started</p>			✓	
5.0 QUALITY CONTROL				
Are samples prepared within the 14-day holding time?				
For each analytical batch of 20 samples or less, are the following quality control samples digested in addition to the field samples?				
- Method blank - reagent water distilled with all reagents				
- Check standard - distilled with all reagents				
- Duplicate sample - separate aliquot of sample				
- Matrix spike - separate aliquot of sample spiked with known concentrations of cyanide and sulfide			✓	

SECTION XIII CYANIDE

METHOD 9010 - TOTAL CYANIDE

Facility Name: Detrex Chemical Industrial, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method is used to determine the concentration of inorganic cyanide (CN ⁻) in aqueous samples. This method is used to quantify the concentration of cyanide from the reactivity test.			✓	The facility lab does not perform this method.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for cyanide analysis?				
Is the SOP available at the analyst's work station?				
Are bound notebooks used to record pertinent data?				
Are notebooks signed and dated by analysts?				
Are notebooks reviewed and signed by a supervisor?				
Are copies of notebook pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available:				
Reactive cyanide apparatus (Section XII)				
Spectrophotometer suitable for measurement at 578 nm with a 1.0-cm cell				
Hot plate stirrer and heating mantle				
pH meter				
Amber light				
Vacuum source				
Refrigerator				
5-ml microburette				
Class A volumetric flasks - 100 and 250 ml				
Erlenmeyer flasks - 500 ml			✓	

SECTION XIII CYANIDE (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better:			✓	
Are the following reagents available for spectrophotometric determination:			✓	
- Sodium hydroxide solution (NaOH) - 0.25N			✓	
- Sodium phosphate monobasic (NaH ₂ PO ₄ · H ₂ O) - 1M			✓	
- Chloramine-T solution (C ₇ H ₇ ClNNaO ₂ S) - 0.44 percent			✓	
- Pyridine-barbituric acid reagent, C ₅ H ₅ N · C ₄ H ₄ N ₂ O ₃			✓	
- Stock potassium cyanide (KCN) solution - 1 ml = 1000 microgram (μg) CN ⁻ (standardized with silver nitrate)			✓	
- Intermediate standard potassium cyanide solution - 1 ml = 100 μg CN ⁻			✓	
- Working standard potassium cyanide solution - 1 ml = 10 μg CN ⁻			✓	
Have the reagents been analyzed for impurities?			✓	
4.0 PROCEDURE The procedure addresses only the manual spectrophotometric determination of total cyanide. The waste should be prepared using the reactivity preparation procedure (Section XII).			✓	
Are 50 ml of the scrubber solution from the reactivity preparation procedure added to a 100-ml volumetric flask?			✓	
Are 15 ml of 1M sodium phosphate solution added and mixed?			✓	
Are 2 ml of chloramine-T solution added and mixed?			✓	
Is the KI-starch paper used to assure an excess of chlorine?			✓	
Are aliquots of chloramine-T solution added to produce an excess of chlorine?			✓	
Are 5 ml of pyridine-barbituric acid solution added and mixed?			✓	
Is reagent water mixed with the sample solution to dilute it to 100 ml?			✓	
Is the sample allowed to stand for 8 minutes for color development?			✓	
Is the absorbance read at 578 nm in a 1-cm cell within 15 minutes?			✓	
Are separate calibration standards prepared for samples with and without sulfides present?			✓	

SECTION XIII CYANIDE (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are working calibration standards made over a concentration range of 0 to 800 $\mu\text{g/L CN}^-$?			✓	
When sulfide is not present in the samples, are at least the high and low standards distilled and analyzed?				
When sulfide is present in the samples, are all standards distilled in the same manner as the samples?				
Is absorbance plotted versus concentration to establish the calibration curve?				
Is the cyanide read off the instrument in mg/L?				
Is the result used to calculate reactive cyanide according to Section XII				
5.0 QUALITY CONTROL				
Are samples prepared within the 14-day holding time?				
Are wastes distilled by using the reactive cyanide procedure (see Section XII)?				
For each analytical batch of 20 samples or fewer, are the following quality control samples digested in addition to the field samples?				
- Method blank - reagent water distilled with all reagents				
- Check standard - distilled with all reagents				
- Duplicate sample - separate aliquot of sample				
- Matrix spike - separate aliquot of sample spiked at 40 $\mu\text{g/L CN}^-$			✓	

SECTION XIV SULFIDE

METHOD 9030 - TOTAL SULFIDE

Facility Name: Detrex Chemical Industrial, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method is used to determine the concentration of hydrogen sulfide (H_2S) in aqueous samples and to quantify the concentration of sulfide from the reactivity test.			✓	The facility lab does not perform perform this method.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for sulfide analysis?				
Is the SOP available at the analyst's work station?				
Are bound notebooks used to record pertinent data?				
Are notebooks signed and dated by analysts?				
Are notebooks reviewed and signed by a supervisor?				
Are copies of notebook pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available:				
Reactive sulfide apparatus (see Section XII)				
pH meter				
5-ml microburette				
Class A volumetric flasks - 100 and 250 ml				
Erlenmeyer flasks - 500 ml				
3.0 REAGENTS All items must be reagent-grade or better.				
Are the following reagents available?				
- Iodine solution - 0.025N solution standardized against phenylarsine oxide or sodium thiosulfate				
- Sodium sulfide nonahydrate ($Na_2S \cdot 9H_2O$) - prepared at suitable concentrations for calibration standards			✓	

SECTION XIV SULFIDE (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
- Titrant - Either 0.025N phenylarsine oxide or 0.025N sodium thiosulfate			✓	
- Hydrochloric acid (HCl) - 6 N			✓	
Have the reagents been analyzed for impurities?				
4.0 PROCEDURE The procedure addresses only the titrimetric determination of total sulfide. The waste should be prepared using the reactivity preparation procedure (see Section XII).				
Is a known amount of standardized iodine solution added to a 500-ml flask, in excess of that needed to oxidize the sulfide, and then brought up to 100 ml with reagent water?				
Is the scrubber solution brought to a pH of 2 with 6N HCl?				
Is the scrubber solution gravimetrically transferred to the 500-ml flask containing the iodine?				
Is the solution in the flask titrated until the amber color is changed to yellow?				
Is starch indicator added to produce a blue color?				
Is the solution titrated until the blue disappears?				
Is the volume of titrant used recorded?				
Is the concentration of sulfide calculated by the following equation? $\text{Sulfide (mg/L)} = \frac{(A * B) - [(C * D) * (32.06 \text{ g/2 eq})]}{E}$ where: A = Amount of iodine solution added (ml) B = Normality of iodine solution C = Amount of titrant used (ml) D = Normality of titrant E = Volume of scrubber solution				
5.0 QUALITY CONTROL				
Are samples prepared within the 14-day holding time?				
Are wastes distilled by using the reactive sulfide procedure (see Section XII)?			✓	

SECTION XIV SULFIDE (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
For each analytical batch of 20 samples or fewer, are the following quality control samples distilled and analyzed in addition to the field samples?			✓	
- Method blank - reagent water distilled with all reagents			↓	
- Check standard - distilled with all reagents			↓	
- Duplicate sample - separate aliquot of sample			↓	
- Matrix spike - separate aliquot of sample spiked			↓	

SECTION XV FLAMMABILITY SCREENING

METHOD - ASTM D4982-89 FLAMMABILITY POTENTIAL SCREENING
ANALYSIS OF WASTES

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method is a qualitative determination of the flammability of slurries, sludges, and solids for treatment and disposal				<p>A modified version of this method is used. A match test is used to determine if the sample is flammable. If so, the Pensky-Martens ignitability test is performed.</p> <p>XV-1 No SOP was available during the audit.</p> <p>* As a result of a formal request, Detrex wrote + submitted an SOP for ignitability tests.</p>
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS. Review the relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for the flammability test?	XV-1*	XV-1		
Is the SOP complete?	✓*		✓	
Is the SOP available at the analyst's work station?			✓	
Is the SOP followed?			✓	
Are bound notebooks use to record pertinent data?		X-1		
Are notebooks signed and dated by analysts?			✓	
Are notebooks reviewed and signed by a supervisor?			✓	
Are copies of notebook pages included in final data package?			✓	
2.0 APPARATUS AND MATERIALS. Are the following apparatus available and in good working order:				
Sample dish - aluminum	✓			
Gas burner and lighter		XV-2		<p>XV-2 The lab used a match to perform flammability screening.</p>
Flint lighter				
Disposable 250-ml plastic beakers				
Metal vessel capable of containing a 250-ml disposable plastic beaker				
Watchglass				
Large pyrex beakers				
Heat-resistant gloves				

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SECTION XV FLAMMABILITY SCREENING (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 PROCEDURE. The procedures are divided into two sections (1) Method A tests the flammability of the waste when exposed to heat and flame; (2) Method B tests the flammability of the waste when exposed to a spark source.				
3.1 Method A - Test Sample Exposed to Heat and Flame				
Is the procedure performed in a draft-free area?	✓			
Are approximately 5 grams of sample placed in an aluminum sample dish?	✓			
Is the flame of the gas burner held immediately above the sample for 2 to 3 seconds?		XV-2		
Is the sample observed for flashing or burning?	✓			
Are the observations recorded?	✓			
Are positively reacting samples further investigated by other methods?	✓			
3.2 Method B - Test Sample Exposed to Spark Source		XV-3		
Are approximately 100 grams of sample added to a plastic disposable beaker?				
Is the beaker and sample placed into the steel vessel, covered with a watchglass, and allowed to stand for 5 minutes?				
Is the ambient temperature recorded?				
Is the watchglass removed and the igniter sparked above the waste?				
Is the combustion condition observed and recorded?		✓		
4.0 QUALITY CONTROL				
If a sample result differs from expected results, does the facility resample and reanalyze?	✓			
Does the laboratory perform duplicate analyses on selected samples?		X-2*		

XV-3 this portion of the test is not performed.

SECTION XVI IGNITABILITY

METHOD 1010 - PENSKEY-MARTENS CLOSED-CUP METHOD

Facility Name: Detrex Chemical Industries, Inc.
 Facility No: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method is used to determine the characteristic of ignitability (for liquids with a flashpoint less than 140°F); the method may be used to characterize waste for treatment or disposal.				
USAGE: Circle the sample types for which this method is used. <u>In-bound waste</u> Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for Method 1010?	XV-1*	XV-1		
Is the SOP available at the analyst's work station?	yes		✓	
Are bound notebooks used to record pertinent data?		X-1		
Are notebooks signed and dated by analysts?			✓	
Are notebooks reviewed and signed by a supervisor?			✓	
Are copies of notebook pages included in the final data package?			✓	
2.0 APPARATUS AND MATERIALS				
Does the laboratory have the appropriate apparatus in good working order?	✓*	XVI-1		
Is proper maintenance performed regularly, including inspection, cleaning, leak tests, and checking and changing of heat-transfer fluid?	✓*		✓	
Is the thermometer NIST certified or checked against an NIST certified thermometer?	✓*		✓	
3.0 REAGENTS All items must be reagent-grade or better.				
Is a suitable calibration fluid available?	✓*	XVI-1		
4.0 PROCEDURE				
Is the testing apparatus cleaned before each sample analysis?	✓*			
Is the testing apparatus checked regularly with a calibration fluid?	✓*			
Is the temperature rise controlled within specified rates?	✓*	↓		

XV-1 During the audit we saw a flashpoint apparatus; however, because the chemist was not available, its use was not fully documented.

In response to a formal request Detrex indicated that the apparatus is used on samples that were determined to be flammable during the screen. An SOP was attached to the response.

SECTION XVI IGNITABILITY (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Does the laboratory use the two-run method, that is, does it determine approximate flashpoint with a rapid temperature rise and well spaced test flames, then obtain definitive results with a new sample aliquot and slow rise in the critical temperature range?		✓		
Is the testing apparatus cleaned after use?	✓*			
5.0 QUALITY CONTROL				
If a sample result differs from expected results, does the facility resample and reanalyze?	✓			
Does the laboratory perform duplicate analyses on selected samples?		X-2*		
Does the laboratory have procedures in place to reevaluate the acceptability of a waste that does not agree with its presumed properties?	✓			
Does the laboratory analyze the calibration fluid regularly?	✓*			

SECTION XVII EPTOX

METHOD 1310 - EXTRACTION PROCEDURE TOXICITY TEST

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: Friday, N. McDermald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method determines whether a waste exhibits the characteristic of toxicity as measured by the extraction procedure (EPTOX). The method simulates the conditions and amount of leaching a waste may undergo if it is disposed of in a sanitary landfill.			✓	The facility lab does not perform this method.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for the EPTOX method?				
Is the SOP available at the analyst's work station?				
Are bound notebooks used to record pertinent data?				
Are notebooks signed and dated by analysts?				
Are notebooks reviewed and signed by a supervisor?				
Are copies of notebook pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean?				
Agitation apparatus				
Extraction vessels				
Filter holder capable of supporting a 0.45-μm filter membrane				
Filter membrane, 0.45 μm				
Prefilters				
Pressure or vacuum filtration device (circle one)				
pH meter accurate to 0.5 unit				
Laboratory balance accurate to 0.01 g				
100-ml beaker for pH aliquots			↓	

SECTION XVII EPTOX (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better.			✓	
Are the following reagents available:			↓	
- Reagent water (with purity verified by analysis for target constituents)				
- Acetic acid, 0.5 N				
- pH buffers at pH 4, 7, and 10				
Have the reagents been analyzed for impurities?				
4.0 PROCEDURE This section is divided into three subsections: preextraction phase separation when free liquid is present, EP when solids are less than 0.5 percent, and EP when solids are greater than 0.5 percent.				
4.1 Preextraction Phase Separation				
If the waste does not contain any free liquid, does the laboratory define the waste as 100 percent solid and omit the preextraction phase separation?				
Does the laboratory weigh the filter membrane and prefilter and record their weights before assembly?				
Are the filter membrane and prefilters assembled in ascending order of pore size?				
Does the facility weigh at least one 100-g sample aliquot?				
Is the liquid filtrate stored in a refrigerated unit until the time of analysis?				
Are oily wastes filtered?				
If the liquid portion of the sample will not pass through the filter (as with heavy oils), is the liquid carried through the EP extraction as a solid?				
Are the filter and residue weighed to 0.01g without drying?				
4.2 Extraction Procedure With Less Than 0.5 Percent Solids				
For samples appearing to be less than 0.5 percent solid, is the percent solid determined precisely?				
Are the filter and residue dried at 80°C to a constant weight?			✓	

SECTION XVII EPTOX (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
If the solid constitutes less than 0.5 percent, are the filter and residue discarded with the filtered liquid used as the extract?			✓	
Is the filtered liquid extract stored in a refrigerator before analysis?				
If the solid constitutes greater than 0.5 percent, are the filter and residue discarded, and is another aliquot taken for extraction?				
4.3 Extraction Procedure With Greater Than 0.5 Percent Solids				
When the percent solids is greater than 0.5 percent, is the net weight of the solid phase (as determined in Section 4.1) used to calculate the amount of water added to the extraction vessel?				
Is the amount of water used to charge the extraction vessel equal to 16 times the sample residue weight?				
Is the pH measured and recorded after agitation has begun?				
If the pH is greater than 5.2, is 0.5N acetic acid solution added to bring the pH to 5.0?				
Is the total amount of acetic acid added less than or equal to 4 ml/g of solid?				
If necessary, is the pH checked and adjusted at regular intervals for the first 6 hours of agitation?				
Is the vessel agitated for 24 hours?				
Is the final pH measured after 24 hours?				
If the final pH is not below 5.2, and the maximum amount of acetic acid has not been added, is the pH adjusted to 5.0 and extraction continued for an additional 4 hours?				
During the additional 4-hour extraction, is the pH measured and recorded at 1-hour intervals?				
At the end of the required extraction time, is water added to the extractor so that the final aqueous portion is equal to 20 times the weight of the solid material used to charge the extractor?				
Is vacuum pressure filtration used to separate the extract from the solids?			✓	

SECTION XVII EPTOX (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Is the filtration stopped when air passes through the membrane or the pressure is 75 pounds per square inch (psi)?			✓	
Are liquids from the initial phase separation and EP extraction combined to form the final EP extract?			↓	
Is the concentration of contaminants for multiphasic extracts determined by using a simple weighted average?			↓	
Is the EP extract preserved and stored for proper analysis?			↓	
5.0 QUALITY CONTROL			↓	
For each analytical batch of 20 samples or fewer, is one blank sample prepared in addition to the field samples?			↓	

SECTION XVIII TCLP

METHOD 1311 - TOXICITY CHARACTERISTIC LEACHING PROCEDURE

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: Chuday / N. McDmald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: The TCLP uses synthetic landfill leachate to extract a waste. For liquid waste, filtered waste is the extract; for biphasic wastes, liquids and solids are separated and handled distinctly. The analysis of the extract is covered in other checklists.			✓	The facility lab does not perform this method.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for TCLP Method 1311?				
Is the SOP available at the analyst's work station?				
Are bound notebooks used to record pertinent data?				
Are notebooks signed and dated by analysts?				
Are notebooks reviewed and signed by a supervisor?				
Are copies of notebook pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean?				
Agitation apparatus (end-over-end at 30 ± 2 rotations per minute)				
Zero-headspace extraction vessel (ZHE) for use with volatile constituents (the vessel has a volume of 500 to 600 ml, a 90- to 100-mm filter, and a pressure gauge available to check for piston tightness and leaks).				
Bottle extraction vessel for use with nonvolatile constituents				
Filter holder for use with nonvolatile constituents				
Filters with pore sizes of 0.6 to 0.8 μm				
pH meter accurate to 0.05 unit				
ZHE extract collection devices (such as Tedlar bags, glass syringes, or similar items) and fluid transfer device (such as peristaltic pumps, syringes, or similar devices)			✓	

SECTION XVIII TCLP (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Laboratory balance accurate to 0.01 g			✓	
500-ml beaker or Erlenmeyer flask with watchglass to cover				
Magnetic stirrer				
3.0 REAGENTS All items must be reagent-grade or better.				
Are the following reagents available?				
- Reagent water - interferant-free water				
- Nitric acid, HNO ₃ - 1N				
- Hydrochloric acid, HCl - 1N				
- Sodium hydroxide, NaOH - 1N				
- Glacial acetic acid				
- Extraction fluid No. 1, sodium acetate buffer (pH 4.93 ± 0.05)				
- Extraction fluid No. 2, dilute acetic acid (pH 2.88 ± 0.05)				
Have the reagents been analyzed for impurities?				
4.0 PROCEDURE This section is divided into three subsections: (1) preliminary procedures that are always used, (2) extraction procedures with no volatiles present, and (3) extraction procedures with volatiles present.				
4.1 Preliminary Evaluation - Determination of Percent Solids				
If no liquid was found in the liquid release test, does the laboratory define the waste as 100 percent solids and omit the determination of percent solids?				
Does the facility preweigh the filter and the receiving vessel for the filtrate before assembling the apparatus?				
Does the facility weigh a 100-g aliquot of sample?				
If the waste is biphasic, is the liquid portion filtered first (centrifugation may be used to speed up settling, but quantitative transfer is essential)?				
After the sample is added to the filtration device (ZHE device for samples with volatile constituents), is vacuum or pressure slowly increased (at 2-minute intervals) until flow stops or pressure reaches 30 psi?			✓	

SECTION XVIII TCLP (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Does the facility define percent solids as $\frac{\text{weight of soil on filter}}{\text{total sample weight}} \times 100$?			✓	
If liquids are trapped in the filter, does the facility dry the filter before weighing it?				
Does the facility have procedures in place to process samples further, as follows?				
• If less than 0.5 percent solids, proceed to nonvolatile or volatile version of the extraction				
• Otherwise, the facility must determine whether particle size reduction is needed				
Does the facility use the criterion of 3.1 (CM ² /g) to determine whether particle size reduction is needed (particles smaller than 3.1 CM ² /g will pass through a 9.5 mm or 0.375-inch sieve)?				
Does the facility determine the appropriate extraction fluid based on one of the following:				
<ul style="list-style-type: none"> • If solids are less than 0.5 percent, no extraction is performed. • For volatile constituents, only extraction fluid No. 1 is used. • Otherwise, 5.0 g of finely divided sample (1 mm in diameter or less) is mixed with 96.5 ml of reagent water for 5 minutes. If the pH is less than 5.0, extraction fluid No. 1 is used. If the pH is greater than 5.0, 3.5 ml 1N HCl is added and heated to 50°C for 10 minutes. If the second extraction has a pH less than 5.0, extraction fluid No. 1 is used. If the pH is equal to or greater than 5.0, extraction fluid No. 2 is used. 				
4.2 Extraction With No Volatile Constituents				
Does the SOP include procedures for carrying out preparation steps, (including separating solids from liquids and reducing particle size) for a sample of at least 100 g when needed?				
Does the laboratory add the solid part of the sample and the proper amount of extraction fluid to the extraction vessel (20 ml/g)?				
Is the sample extracted under rotary agitation for 18 ± 2 hour at 22 ± 3°C?			✓	

SECTION XVIII TCLP (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Does the facility filter the sample through a new glass fiber filter and reserve the filtrate for analysis?			✓	
Is the filtrate pH checked and recorded?				
Is the aliquot of the filtrate intended for metals analyses acidified with HNO ₃ to below a pH of 2 (If acidification causes precipitation, is preservation omitted and analyses performed as soon as possible)?				
4.3 Extraction With Volatile Constituents				
Does the facility preweigh the filtrate collection container?				
Is the ZHE device properly assembled?				
Is the appropriate amount of sample placed in the ZHE device? If less than 0.5 percent solids, does the laboratory use 500 g; otherwise, does the facility determine the weight in grams according to the following equation: $\text{weight} = \frac{25}{\text{percent solids}} \times 100$				
Is particle size reduction performed when necessary in a manner that minimizes loss by volatilization?				
Is the ZHE device lightly pressurized to eliminate all headspace?				
Is the pressure then slowly increased to remove the liquid phase?				
Is the liquid phase analyzed immediately or properly stored until analysis?				
Is the proper amount of extraction fluid No. 1 added to the ZHE according to the following equation? $\text{Amount of fluid} = \frac{20 \times \text{weight of solids in ZHE}}{100} \text{ (grams)}$				
Is the vessel leak tested to ensure that it is lightly pressurized and then properly assembled in the agitation apparatus and run for 18 ± 2 hours at 23 ± °C?				
After the run, is the ZHE vessel pressure checked for consistency with the initial pressure?				
If leaks are detected, is the extraction restarted on a new sample aliquot?			✓	

SECTION XVIII TCLP (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Is the filtrate separated from the solid phase and prepared for analysis immediately?			✓	
5.0 QUALITY CONTROL				
Are holding times monitored by analysts?				
Does the facility have adequate means for handling difficult samples (such as multiphasic or viscous liquids)?				
Are extracts preserved and stored consistently with the appropriate analytical method?			✓	

SECTION XIX SPECIFIC GRAVITY

METHOD - Detrex SOP

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This method measures the specific gravity of a sample by using hydrometers; the method is used to estimate the solvent content of the sample.				
USAGE: Circle the sample types for which this method is used. <u>In-bound waste</u> Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for the specific gravity determination?	✓			
Is the SOP available at the analyst's work station?	✓			
Are bound notebooks used to record pertinent data?		X-1		
Are notebooks signed and dated by analysts?			✓	
Are notebooks reviewed and signed by a supervisor?			✓	
Are copies of notebook pages included in the final data package?			✓	
2.0 APPARATUS AND MATERIALS Are the following apparatus available and in good working order?				
Hydrometers, with the glass graduated in appropriate units	✓			
Hydrometer jar	✓			
Thermometers	✓			
3.0 PROCEDURE				
Is the temperature of the hydrometer cylinder brought to about the same temperature as the sample to be tested?	✓			
Is the hydrometer lowered into the sample so that the stem is not wet above the level it will be immersed in the liquid?	✓			
Is the sample continuously stirred with the thermometer?	✓			
Is the temperature recorded to the nearest 0.25°C?	✓			
Is the hydrometer depressed about two scale divisions into the liquid, then released?	✓			

SECTION XIX SPECIFIC GRAVITY (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
After the hydrometer has come to a rest, is the scale read to the nearest 0.01 specific gravity unit and recorded?	✓			
Is the solvent percent calculated from the Solvent-Oil Mixture vs. Gravity Chart in the SOP?	✓			
4.0 QUALITY CONTROL				
Is this procedure performed in a draft-free area?	✓			
If a sample result differs from expected results, does the facility resample and reanalyze?	✓			
Does the laboratory perform duplicate analyses on selected samples?		X-2*		

SECTION XX SPENT SOLVENT EXTRACTION

METHOD - Detrex SOP

(Rotovap Distillation)

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Friday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This is an organic extraction method used to prepare spent solvent oil samples to determine volatile solvent content. The sample is distilled and the total nonvolatile percentage is calculated.				
USAGE: Circle the sample types for which this method is used. <input checked="" type="checkbox"/> In-bound waste <input type="checkbox"/> Process control <input type="checkbox"/> Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for solvent extraction?	✓			
Is the SOP available at the analyst's work station?	✓			
Are analysis runlogs used to record pertinent data?		X-1		
Are runlogs signed and dated by analysts?			✓	
Are runlogs reviewed and signed by a supervisor?			✓	
Are copies of runlog pages included in the final data package?			✓	
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean?				
- Rotary vacuum evaporator with heating bath	✓			
- 250-ml flat-bottom flasks	✓			
- Analytical balance capable of measuring to 0.005 g	✓			
- Vacuum apparatus consisting of a water aspirator	✓			
3.0 PROCEDURE				
Is a 30-g aliquot of sample measured into a tared flat-bottom flask?	✓			
Is the precise weight recorded?	✓			
Is the flask attached to the rotary vacuum evaporator and lowered into a 90°C water bath?	✓			
Is the sample allowed to distill until condensate ceases to be produced?	✓			

SECTION XX SPENT SOLVENT EXTRACTION (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Is the residue in the flask weighed and recorded?	✓			
Is the percent of nonvolatile residue calculated with the following equation: $\%NV = (R/S) \times 100$ where: $\%NV$ = Percent nonvolatile residue R = Mass of residue after roto-evaporation S = Mass of spent solvent before roto-evaporation	✓			
4.0 QUALITY CONTROL				
If a sample result differs from expected results, does the facility resample and reanalyze?	✓			
Does the laboratory perform duplicate analyses on selected samples?		X-2*		

SECTION XXI SOLVENT CONTENT

METHOD - Detrex SOP

Facility Name: Detrex Chemical Industries, Inc.Facility ID No.: MID 691 605 972Reviewer: C. Friday, J. N. McDonaldDate Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This GC method is used to measure solvent content by direct injection; detection is accomplished with a flame ionization detector (FID).				
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for determining solvent content by GC?	✓			
Is the SOP available at the analyst's work station?	✓			
Are analysis runlogs used to record pertinent data?		XXI-1		
Are runlogs signed and dated by analysts?			✓	
Are runlogs reviewed and signed by a supervisor?			✓	
Are copies of runlog pages included in the final data package?			✓	
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean:				
Gas chromatograph	✓			
Flame ionization detector	✓			
Chromatography columns	✓			
Data systems		XXI-2		
Syringes, 5 mL	✓			
Microsyringes, 10, 25, and 100 µL	✓			
Class A volumetric flasks with ground glass stopper: 10, 50, 100, 500, and 1,000 mL	✓			
Analytical balance sensitive to 0.1 mg	✓			

XXI-1 Results are recorded on the laboratory ~~data~~^{report} results summary form for each sample. Chromatograms are attached to the form.

XXI-2 The lab had an integrator only.

SECTION XXI SOLVENT CONTENT (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better.				
Are the following reagents available:				
- Reagent water, organic-free		VI-2*		
- Chlorobenzene solvent	✓			
- Stock standards for halogenated and nonhalogenated solvents, purchased as pure chemicals and prepared according to appropriate SOPs	✓			
- At least twice calibration standards, including one near the required quantitation limit, prepared from secondary dilution standards	✓			
Are fresh standards prepared as follows:				
- Every 2 months for gases and reactive compounds			✓	
- Every 6 months for all others	✓ *			
- As needed, if check standards imply a problem	✓			
Have the reagents been analyzed for impurities?	✓			
4.0 PROCEDURES				
Are high-concentration liquids directly injected (10 µL or similar amount for detection limits of 10,000 µg/L or higher)?	✓			
Are samples screened (by Method 3810, automated headspace sampler, or Method 3820, hexadecane extraction)?			✓	
Are samples and standard solutions allowed to warm to ambient temperatures before analysis?	✓			
Is the sample properly diluted, if required?	✓			
Are the following GC conditions or their demonstrated equivalents used?				
- Helium, 180 psi	✓			
- Initial temperature, 35°C for 6 min	✓			
- Raise 4°C per min to 90°C	✓			

SECTION XXI SOLVENT CONTENT (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
- Raise 10°C per min to 180°C	✓			
- Hold at 180°C for 1 min	✓			
Does the laboratory use the same column introduction technique for calibration and for samples?	✓			
Does the laboratory use three standards covering the expected working range?	✓			
Are the correction factors (CF) and response factors (RF) properly calculated as follows: Note: External Standards, $CF = \frac{\text{Peak Area}}{\text{percent component}}$ Internal Standards, $RF = \frac{A_s C_{is}}{A_{is} C_s}$ where: A_s = Area of calibration standard A_{is} = Area of internal standard C_s = Concentration of calibration standard C_{is} = Concentration of internal standard	✓			
Is the relative standard deviation (RSD) of the CF or RF calculated?	✓			
If RSD exceeds 20 percent, does the laboratory assume nonlinearity and make proper corrections?	✓			
Does the laboratory determine retention time windows as three times the standard deviation of the absolute retention times of three standards injected over a 72-hour period?	✓*			
Does each day's run begin with a reagent blank and a midconcentration standard?	✓*			
If percent difference of RF or CF differs more than 15 percent from the initial calibration, does the laboratory begin corrective action (such as inspection of systems, maintenance as needed, and new initial calibration if required)?	✓			
Does the laboratory establish daily retention windows with the midpoint determined from continuing calibration and width from initial calibration?	✓*			

SECTION XXI SOLVENT CONTENT (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Is an additional continuing calibration run after every 10 samples and at the end of the day?		XXI -3		XXI-3 The continuing calibration is analyzed after 30 samples instead of 10.
Does the laboratory have procedures for dilutions when a result exceeds the calibration range?	✓			
Does the laboratory use periodic laboratory control samples, quality check samples, performance evaluation samples, or similar results?	✓			
Does the laboratory use at least one matrix spike per analytical batch?	✓			
Does the laboratory use at least one matrix duplicate or matrix spike duplicate per analytical batch?	✓			
Is the following formula used to calculate solvent concentrations: Concentration (% wt/vol) = $(A_x / CF) \times D$ where: A_x = Analyte response (peak area) CF = Calibration factor D = Dilution factor	✓			
Does the laboratory check calculations, whether they are performed manually or automatically by computer?	✓			
Are undetected results adjusted properly for dilutions?	✓			
5.0 QUALITY CONTROL				
Are samples analyzed within the 14-day holding time?	✓			
Does the laboratory have procedures to start corrective actions when quality control measures (such as matrix spikes, duplicates, laboratory control standards, and surrogates) are out of limits?	✓			
Is the analysis report produced, checked, and forwarded to the appropriate person before action is taken on the waste?	✓			

SECTION XXII HALOGENATED SOLVENTS

METHOD 8010 - HALOGENATED VOLATILE ORGANICS ANALYSIS

Facility Name: Detrex Chemical Industries, Inc.Facility ID No.: MID 091 605 972Reviewer: Friday, N. McDonaldDate Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This GC method is used for halogenated organics analysis and uses either direct injection or a purge-and-trap device (Method 5030). Detection is accomplished with an electrolytic conductivity detector.			✓	This method is not performed by the facility's lab.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for Method 8010?				
Is the SOP available at the analyst's work station?				
Are analysis runlogs used to record pertinent data?				
Are runlogs signed and dated by analysts?				
Are runlogs reviewed and signed by a supervisor?				
Are copies of runlog pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean?				
- Gas chromatograph with electrolytic conductivity detector				
- Purge-and-trap device				
- Chromatography columns				
- Data systems				
- Syringes, 5 ml				
- Microsyringes, 10, 25, and 100 µL				
- Class A volumetric flasks with ground glass stopper: 10, 50, 100, 500, and 1,000 ml				
- Analytical balance sensitive to 0.1 mg			✓	

SECTION XXII HALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better.			✓	
Are the following reagents available:			↓	
- Reagent water, organic-free			↓	
- Methanol (pesticide quality)			↓	
- Stock standards purchased as pure chemicals and prepared in methanol in accordance with appropriate SOPs			↓	
- Stock standards purchased as certified solutions			↓	
- Secondary dilution standards			↓	
- At least five calibration standards, including one near the required quantitation limit, prepared from secondary dilution standards			↓	
- Internal standards (if used for calibration)			↓	
- Surrogate standards (bromochloromethane, bromochlorobenzene, and bromofluorobenzene are recommended).			↓	
Are the standards prepared freshly as follows:			↓	
- Every 2 months for gases and reactive compounds			↓	
- Every 6 months for all others			↓	
- As needed, if check standards imply a problem			↓	
Have the reagents been analyzed for impurities?			↓	
4.0 PROCEDURES Procedures consist of sample introduction, GC conditions, analytical run, and calculations. Most procedures are standard GC from Method 8000.			↓	
4.1 Sample Introduction			↓	
In all methods, are surrogate standards and internal standards (if used) added and mixed before introduction?			↓	
Are high-concentration liquids directly injected (10 µL or similar amount for detection limits of 10,000 µg/L or higher)?			✓	

SECTION XXII HALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are samples screened (by Method 3810, automated headspace sampler, and by Method 3820, hexadecane extraction)?			✓	
Are samples and standard solutions allowed to warm to ambient temperatures before analysis?				
Is the purge-and-trap device assembled correctly? Note that absorbents consist of one-third 2,6-diphenylene oxide (at inlets); one-third silica gel; and one-third charcoal; with optional 1.0 cm of methyl silicone-coated packing at the inlet.				
Is the sample properly diluted, if required?				
Are high-concentration solids extracted with methanol (that is, a methanol aliquot is added to water and treated as a water sample)?				
Are low-concentration solids mixed with water in the device?				
4.2 GC Conditions Are the following conditions or their demonstrated equivalents used?				
Purge-and-trap device (if used):				
- Nitrogen or helium at 40 mL/min to purge				
- 11.0 ± 0.1 min at ambient purging				
- Desorb 4 min at 180°C				
- 20 to 60 mL/min backflush inert gas flow				
Gas Chromatography - Column 1:				
- Helium flow, 40 mL/min				
- Initial temperature, 45°C for 3 min				
- Raise 8°C per min to 220°C				
- Hold at 220°C for 15 min				
Gas Chromatography - Column 2:				
- Helium flow, 40 mL/min				
- Initial temperature, 50°C for 3 min			✓	

SECTION XXII HALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
- Raise 6°C per min to 170°C			✓	
- Hold at 170°C for 4 min			✓	
4.3 Analytical Run				
Does the laboratory use the same column introduction technique for calibration and for samples?				
Does the laboratory use at least five standards covering the expected working range?				
Does each calibration standard include the surrogates and (if used) internal standards used for samples?				
Are the correction factors (CF) and response factors (RF) properly calculated as follows: Note: External Standards, $CF = \frac{\text{Peak Area}}{\text{nanogram injected}}$ Internal Standards, $RF = \frac{A_s C_{is}}{A_{is} C_s}$ where: A_s = Area of calibration standard A_{is} = Area of internal standard C_s = Concentration of calibration standard C_{is} = Concentration of internal standard				
Is the relative standard deviation (RSD) of the CF or RF calculated?				
If RSD exceeds 20 percent, does the laboratory assume nonlinearity and make proper corrections?				
Does the laboratory determine retention time windows as three times the standard deviation of the absolute retention times of three standards injected over a 72-hour period?				
Do daily runs begin with a reagent blank and a midconcentration standard?			✓	

SECTION XXII HALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
If percent difference of RF or CF differs more than 15 percent from the initial calibration, does the laboratory begin corrective action (such as inspection of systems, maintenance as needed, and new initial calibration if required)?			✓	
Does the laboratory establish daily retention windows with the midpoint determined from continuing calibration and width from initial calibration?				
Is an additional continuing calibration run after every 10 samples and at the end of the day?				
Does the laboratory have procedures for dilutions when a result exceeds the calibration range?				
Does the laboratory use periodic laboratory control samples, quality check samples, performance evaluation samples, or similar results?				
Does the laboratory use at least one matrix spike per analytical batch?				
Does the laboratory use at least one matrix duplicate or matrix spike duplicate per analytical batch?				
4.4 Calculations				
Are the following formulas and calculations used for the analyses?				
<p>External Standards, Aqueous:</p> <p>Concentration ($\mu\text{g/L}$) = $\frac{A_x A V_e D}{A_s V_i V_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height)</p> <p>A = Amount of standard, nanograms (ng)</p> <p>V_e = Volume of total extract, μL</p> <p>D = Dilution factor</p> <p>A_s = Standard response (units of A_x)</p> <p>V_i = Volume of extract injected, μL (for purge-and-trap, $V_e = V_i = 1$)</p> <p>V_s = Volume of sample, mL</p>			↓	

SECTION XXII HALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
<p>External Standards, Nonaqueous:</p> <p>Concentration ($\mu\text{g/kg}$) = $\frac{A_x A V_e D}{A_s V_i W_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height) A = Amount of standard, nanograms (ng) V_e = Volume of total extract, μL D = Dilution factor A_s = Standard response (units of A_x) V_i = Volume of extract injected, μL (for purge-and-trap, $V_e = V_i = 1$) W_s = Weight of sample, g</p>			✓	
<p>Internal Standards, Aqueous:</p> <p>Concentration ($\mu\text{g/L}$) = $\frac{A_x C_i D}{A_{is} RF V_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height) C_{is} = Amount of internal standard, ng D = Dilution factor A_{is} = Internal standard response (units of A_x) RF = Response factor V_s = Volume of sample injected, μL</p>			✓	

SECTION XXIII NONHALOGENATED SOLVENTS

METHOD 8015 - NONHALOGENATED VOLATILE ORGANICS ANALYSIS

Facility Name: Detrex Chemical Industries, Inc.
 Facility ID No.: MID 091 605 972
 Reviewer: C. Fuday / N. McDonald
 Date Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This GC method is used for nonhalogenated organics analysis and uses either direct injection or a purge-and-trap device (Method 5030). Detection is accomplished with a flame ionization detector (FID)			✓	This method is not performed by the facility's lab.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for Method 8015?				
Is the SOP available at the analyst's work station?				
Are analysis runlogs used to record pertinent data?				
Are runlogs signed and dated by analysts?				
Are runlogs reviewed and signed by a supervisor?				
Are copies of runlog pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean?				
Gas chromatograph with FID				
Purge-and-trap device				
Chromatography columns				
Data system				
Syringes, 5 ml				
Microsyringes, 10, 25, and 100 µl				
Class A volumetric flasks with ground glass stopper: 10, 50, 100, 500, and 1,000 ml				
Analytical balance, sensitive to 0.1 mg			✓	

SECTION XXII HALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Internal Standards, Nonaqueous: Concentration ($\mu\text{g/kg}$) = $\frac{A_x C_{is} D}{A_{is} RF W_s}$ where: A_x = Analyte response (area or height) C_{is} = Amount of internal standard, ng D = Dilution factor A_{is} = Internal standard response (units of A_x) RF = Response factor W_s = Weight of sample, g				
(Optional) Conversion to Dry Weight: Concentration, dry weight = <u>Analytical Result (wet weight)</u> (percent solids)				
Does the laboratory check calculations whether they are performed manually or automatically by computer?				
Are undetected results adjusted properly for dilutions?				
5.0 QUALITY CONTROL				
Does the laboratory have procedures for starting corrective actions when quality control measures (such as matrix spikes, duplicates, laboratory control standards, and surrogates) are out of limits?				
Is the analysis report produced, checked, and forwarded to the appropriate person before action is taken on the waste?				

SECTION XXIII NONHALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better.			✓	
Are the following reagents available:				
- Reagent water, organic-free				
- Methanol (pesticide quality)				
- Stock standards, purchased as pure chemicals and prepared in methanol by appropriate SOP				
- Stock standards, purchased as certified solutions				
- Secondary dilution standards				
- Calibration standards; at least five, including one near MDL, prepared from secondary dilution standards				
- Internal standards (if used for calibration)				
- Surrogate standards				
Are the standards prepared freshly as follows:				
- Every 2 months for gases and reactive compounds				
- Every 6 months for all others				
- As needed, if check standards imply a problem				
Have the reagents been analyzed for impurities?				
4.0 PROCEDURES Procedures consist of sample introduction, GC conditions, procedure, and calculations. Most procedures are standard GC methods from Method 8000.				
4.1 Sample Introduction				
In all methods, are surrogate standards and internal standards (if used) added and mixed before introduction?				
Are high-concentration liquids directly injected (10 µL or similar amount for detection limits 10,000 µg/L or higher)?			✓	

SECTION XXIII NONHALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are low concentration liquids introduced by a purge-and-trap device (Method 5030) as follows?			✓	
Are samples screened (by Method 3810, automated headspace sampler, and by Method 3820, hexadecane extraction)?				
Are samples and standard solutions warmed to ambient temperatures before analysis?				
Is the purge-and-trap device assembled correctly? Note that absorbents consist of one-third 2,6-diphenylene oxide (at inlet); one-third silica gel; and one-third charcoal; with optional 1.0 cm of methyl silicone-coated packing at the inlet.				
Are samples properly diluted, if required?				
Are high-concentration solids extracted with methanol (that is, a methanol aliquot is added to water and treated as a water sample)?				
4.2 GC Conditions Are the following conditions or their demonstrated equivalents used:				
Purge-and-trap device				
- Nitrogen or helium at 20 mL/min to purge				
- 15.0 ± 0.1 min at 85 ± 2°C purging				
- Desorb 1.5 min at 180°C				
- 20 to 60 mL/min backflush inert gas flow				
Gas Chromatography - Column 1				
- Helium flow, 40 mL/min				
- Initial temperature, 45°C for 3 min				
- Raise 8°C/min to 220°C				
- Hold at 220°C* for 15 min				
Gas Chromatography - Column 2				
- Helium flow, 40 mL/min				
- Initial temperature, 50°C for 3 min			✓	

SECTION XXIII NONHALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
- Raise 6°C/min to 170°C			✓	
- Hold at 170°C for 4 min				
4.3 Procedure				
Does the laboratory use the same column introduction technique for calibration and for samples?				
Does the laboratory use at least five standards covering the expected working range?				
Does each calibration standard include the surrogates and (if used) internal standards used for samples?				
Are the correction and response factors (CF and RF) properly calculated as follows: <div style="margin-left: 40px;"> <p>Note: External Standards, $CF = \frac{\text{Peak Area}}{\text{Nanogram injected}}$</p> <p>Internal Standards, $RF = \frac{A_s C_{is}}{A_{is} C_s}$</p> <p>where:</p> <p>$A_s$ = Area of calibration standard</p> <p>A_{is} = Area of internal standard</p> <p>C_s = Concentration of calibration standard</p> <p>C_{is} = Concentration of internal standard</p> </div>				
Is the relative standard deviation (RSD) of the CF or RF calculated?				
If RSD exceeds 20 percent, does the laboratory assume nonlinearity and make proper corrections?				
Does the laboratory determine retention time windows as three times the standard deviation of the absolute retention times of three standards injected over a 72-hour period?				
Does each daily run begin with a reagent blank and a midcalibration standard?			✓	

SECTION XXIII NONHALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
If percent difference of RF or CF differs by more than 15 percent from the initial calibration, does the laboratory begin corrective action (such as inspection of systems, maintenance as needed, and new initial calibration if required)?			✓	
Does the laboratory establish daily retention windows with the midpoint determined from continuing calibration and width from initial calibration?				
Is an additional continuing calibration run after every 10 samples and at the end of the day?				
Does the laboratory have procedures for dilutions to be used when a result exceeds the calibration range?				
Does the laboratory use periodic laboratory control samples, quality check samples, performance evaluation samples, or similar results?				
Does the laboratory use at least one matrix spike per analytical batch?				
Does the laboratory use at least one matrix duplicate or matrix spike duplicate per analytical batch?				
4.4 Calculations				
Are the following formulas and calculations used for the analyses?				
<p>External Standards, Aqueous:</p> <p>Concentration ($\mu\text{g/L}$) = $\frac{A_x A V_e D}{A_s V_i V_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height)</p> <p>A = Amount of standard, ng</p> <p>V_e = Volume of total extract, μL</p> <p>D = Dilution factor</p> <p>A_s = Standard response (units of A_x)</p> <p>V_i = Volume of extract injected, μL (for purge-and-trap, $V_e = V_i = 1$)</p> <p>V_s = Volume of sample, mL</p>			↓	

SECTION XXIII NONHALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
<p>External Standards, Nonaqueous:</p> <p>Concentration ($\mu\text{g/kg}$) = $\frac{A_x A V_c D}{A_s V_i W_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height) A = Amount of standard, ng V_c = Volume of total extract, μL D = Dilution factor A_s = Standard response (units of A_x) V_i = Volume of extract injected, μL (for purge-and-trap, $V_c = V_i = 1$) W_s = Weight of sample, g</p>			✓	
<p>Internal Standards, Aqueous:</p> <p>Concentration ($\mu\text{g/L}$) = $\frac{A_x C_{is} D}{A_{is} RF V_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height) C_{is} = Amount of internal standard, ng D = Dilution factor A_{is} = Area of internal standard RF = Response factor V_s = Volume of sample used, mL</p>			✓	
<p>Internal Standards, Nonaqueous:</p> <p>Concentration ($\mu\text{g/kg}$) = $\frac{A_x C_{is} D}{A_{is} RF W_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height) C_{is} = Amount of internal standard, ng D = Dilution factor A_{is} = Area of internal standard RF = Response factor W_s = Volume of sample used, g</p>			✓	

SECTION XXIII NONHALOGENATED SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
(Optional) Conversion to Dry Weight: Concentration, dry weight = $\frac{\text{Analytical Results (wet weight)}}{\text{(percent solids)}}$			✓	
Does the laboratory check calculations whether they are performed manually or automatically by computer?			↓	
Are undetected results adjusted properly for dilutions?			↓	
5.0 QUALITY CONTROL			↓	
Does the laboratory have procedures to start corrective actions when quality control measures (such as matrix spikes, duplicates, laboratory control standards, and surrogates) are out of limits?			↓	
Is the analysis report produced, checked, and forwarded to the appropriate person before action is taken on the waste?			↓	

SECTION XXIV AROMATIC SOLVENTS

METHOD 8020 - AROMATIC VOLATILE ORGANICS ANALYSIS

Facility Name: Detrex Chemical Industries, Inc.Facility ID No.: MID 091 605 972Reviewer: C. Friday / N. McDonaldDate Completed: 5-18-95

Requirements	Yes	No	NA	Comments (Explain all negative responses)
SUMMARY: This GC method is used for aromatic organics analysis and uses either direct injection or a purge-and-trap device (Method 5030). Detection is accomplished with an photoionization detector (PID).			✓	This method is not performed by the facility lab.
USAGE: Circle the sample types for which this method is used. In-bound waste Process control Out-bound waste				
1.0 DOCUMENTS Review relevant SOPs and laboratory documents as follows:				
Does the facility have an SOP for Method 8020?				
Is the SOP available at the analyst's work station?				
Are analysis runlogs used to record pertinent data?				
Are runlogs signed and dated by analysts?				
Are runlogs reviewed and signed by a supervisor?				
Are copies of runlog pages included in the final data package?				
2.0 APPARATUS AND MATERIALS Are the following items available in good repair and clean?				
Gas chromatograph with PID				
Purge-and-trap device				
Chromatography columns				
Data system				
Syringes, 5 ml				
Microsyringes, 10, 25, and 100 µl				
Class A volumetric flasks with ground glass stopper: 10, 50, 100, 500, and 1,000 mL				
Analytical balance, sensitive to 0.1 mg			✓	

SECTION XXIV AROMATIC SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
3.0 REAGENTS All items must be reagent-grade or better.			✓	
Are the following reagents available?			✓	
- Reagent water, organic-free			✓	
- Methanol (pesticide quality)			✓	
- Stock standards, purchased as pure chemicals and prepared in methanol in accordance with the appropriate SOP.			✓	
- Stock standards, purchased as certified solutions			✓	
- Secondary dilution standards			✓	
- At least five calibration standards, including one near required quantitation limit, prepared from secondary dilution standards			✓	
- Internal standards (if used for calibration)			✓	
- Surrogate standards (bromochloromethane; bromochlorobenzene; 1,1,1-trifluorotoluene; fluorobenzene; and difluorobenzene are recommended)			✓	
Are the standards prepared freshly as follows:			✓	
- Every 2 months for gases and reactive compounds			✓	
- Every 6 months for all others			✓	
- As needed, if check standards imply a problem			✓	
Have the reagents been analyzed for impurities?			✓	
4.0 PROCEDURES Procedures consist of sample introduction, GC conditions, procedure, and calculations. Most of these are standard GC methods from Method 8000.			✓	
4.1 Sample Introduction			✓	
In all methods, are surrogate standards and internal standards (if used) added and mixed before introduction?			✓	
Are high-concentration liquids directly injected (10 µL or similar amount for detection limits 10,000 µg/L or higher)?			✓	

SECTION XXIV AROMATIC SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Are samples screened (by Method 3810, automated headspace sampler, and by Method 3820, hexadecane extraction)?			✓	
Are samples and standard solutions allowed to warm to ambient temperatures before analysis?				
Is the purge-and-trap device assembled correctly? Note that absorbents consist of one-third 2,6-diphenylene oxide (at inlet), one-third silica gel, and one-third charcoal, with optional 1.0 cm of methyl silicone-coated packing at the inlet?				
Is the sample properly diluted, if required?				
Are high-concentration solids extracted with methanol (that is, a methanol aliquot is added to water and treated as a water sample)?				
Are low-concentration solids mixed with water in the device?				
4.2 GC Conditions Are the following conditions or their demonstrated equivalent used?				
Purge-and-trap device (if used)				
- Nitrogen or helium at 40 mL/min to purge				
- 11.0 ± 0.1 min at ambient purging				
- Desorb 4 min at 180°C				
- 20- to 60-mL/min backflush inert gas flow				
Gas Chromatography - Column 1 (primary column)				
- Helium flow, 36 mL/min				
For lower boiling compounds:				
- Initial temperature, 50°C for 2 min				
- Raise 6°C/min to 90°C				
- Hold at 90°C until all compounds have eluted				
For higher boiling compounds:				
- Initial temperature, 50C for 2 min			✓	

SECTION XXIV AROMATIC SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
- Raise 3°C/min to 110°C			✓	
- Hold at 110°C until all compounds have eluted				
Gas Chromatography - Column 2 (confirmatory column)				
- Helium flow, 30 mL/min				
- Initial temperature, 40°C for 2 min				
- Raise 2°C/min to 100°C				
- Hold at 100°C until all compounds have eluted				
4.3 Procedure				
Does the laboratory use the same column introduction technique for calibration and for samples?				
Does the laboratory use at least five standards covering the expected working range?				
Does each calibration standard include the surrogates and (if used) internal standards used for samples?				
Are the correction and response factors (CF and RF) properly calculated as follows: <div style="margin-left: 40px;"> External Standards, CF = $\frac{\text{Peak Area}}{\text{nanogram injected}}$ Internal Standards, RF = $\frac{A_s C_{is}}{A_{is} C_s}$ </div> where: <div style="margin-left: 40px;"> A_s = Area of calibration standard C_{is} = Concentration of internal standard A_{is} = Area of internal standard C_s = Concentration of calibration standard </div>				
Is the relative standard deviation (RSD) of the CF or RF calculated?				
If RSD exceeds 20 percent, does the laboratory assume nonlinearity and make proper corrections?			✓	

SECTION XXIV AROMATIC SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
Does each daily run begin with a reagent blank and midconcentration standard?			✓	
If percent difference of RF or CF differs by more than 15 percent from the initial calibration, does the laboratory begin corrective action (such as inspection of systems, maintenance as needed, and new initial calibration if required)?				
Does the laboratory establish daily retention windows with the midpoint determined from continuing calibration and width from initial calibration?				
Is an additional continuing calibration run after every 10 samples and at the end of the day?				
Does the laboratory have procedures for dilutions when a result exceeds the calibration range?				
Does the laboratory use periodic laboratory control samples, quality check samples, performance evaluation samples, or similar results?				
Does the laboratory use at least one matrix spike per analytical batch?				
Does the laboratory use at least one matrix duplicate or matrix spike duplicate per analytical batch?				
4.4 Calculations				
Are the following formulas and calculations used for the analyses?				
<p>External Standards, Aqueous:</p> <p>Concentration ($\mu\text{g/L}$) = $\frac{A_x A V_c D}{A_s V_i V_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height)</p> <p>A = Amount of standard, ng</p> <p>V_c = Volume of total extract, μL</p> <p>D = Dilution factor</p> <p>A_s = Standard response (units of A_x)</p> <p>V_i = Volume of extract injected, μL (for purge-and-trap, $V_e = V_i = 1$)</p> <p>V_s = Volume of sample, mL</p>			↓	

SECTION XXIV AROMATIC SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
<p>External Standards, Nonaqueous:</p> <p>Concentration ($\mu\text{g/kg}$) = $\frac{A_x A V_e D}{A_s V_i W_s}$</p> <p>where:</p> <p>$A_x$ = Analyte response (area or height) A = Amount of standard, ng V_e = Volume of total extract, μL D = Dilution factor A_s = Standard response (units of A_x) V_i = Volume of extract injected, μL W_s = Weight of sample, g</p>			✓	
<p>Internal Standards, Aqueous:</p> <p>Concentration ($\mu\text{g/L}$) = $\frac{A_x C_{is} D}{A_{is} RF V_s}$</p> <p>where:</p> <p>$A_x$ = Area of calibration standard C_{is} = Amount of internal standard, ng D = Dilution factor A_{is} = Response of internal standard RF = Response factor V_s = Amount of sample injected</p>			✓	
<p>Internal Standards, Nonaqueous:</p> <p>Concentration ($\mu\text{g/kg}$) = $\frac{A_x C_{is} D}{A_{is} RF W_s}$</p> <p>where:</p> <p>$A_x$ = Area of calibration standard C_{is} = Amount of internal standard, ng D = Dilution factor A_{is} = Response of internal standard RF = Response factor W_s = Weight of sample, g</p>			✓	

SECTION XXIV AROMATIC SOLVENTS (continued)

Requirements	Yes	No	NA	Comments (Explain all negative responses)
(Optional) Conversion to dry weight: Concentration, dry weight = $\frac{\text{Analytical Result (wet weight)}}{\text{(Percent solids)}}$			✓	
Does the laboratory check calculations whether they are done manually or automatically by computer?			↓	
5.0 QUALITY CONTROL				
Does the laboratory have procedures for starting corrective actions when quality control measures (such as matrix spikes, duplicates, laboratory control standards, and surrogates) are out of limits?			↓	
Is the analysis report produced, checked, and forwarded to the appropriate person before action is taken on the waste?			↓	

SECTION XXV REFERENCES

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FINAL
RCRA LABORATORY COMPLIANCE EVALUATION INSPECTION REPORT

DETREX CHEMICAL INDUSTRIES, INC.
DETROIT, MICHIGAN
MID 091 605 972

Prepared for:

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EPA Region	:	5
Date Prepared	:	March 20, 1996
Contract	:	68-W4-0007
PRC Project Manager	:	Candace Friday
Telephone	:	713/589-6477
EPA Work Assignment Manager	:	Ann E. Kerbs
Telephone	:	312/886-3901

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Attachment

RCRA LABORATORY COMPLIANCE EVALUATION INSPECTION CHECKLIST

1.0 INTRODUCTION

PRC Environmental Management, Inc. (PRC), received Work Assignment No. R05037 from the U.S. Environmental Protection Agency (EPA) under Contract No. 68-W4-0007. This work assignment involves performing seven laboratory audits as part of state-lead compliance evaluation inspections of in-house laboratories that perform waste analyses at treatment, storage, and disposal facilities throughout the state of Michigan. The laboratory audits were performed by PRC under the authority of EPA and Section 3007 of the Resource Conservation and Recovery Act (RCRA), 42 U.S.C. Section 6927, and under the lead of Michigan Department of Natural Resources (MDNR) district inspectors. This document discusses the laboratory audit findings for Detrex Chemical Industries, Inc. (Detrex), of Detroit, Michigan.

Before conducting the on-site laboratory audit, PRC reviewed the facility waste analysis plan (Detrex 1991a) and prepared detailed, facility-specific laboratory checklists. The checklists consist of questions taken directly from Appendix A of the EPA document *RCRA Laboratory Audit Inspection Guidance Document* (EPA 1988) as well as specific procedural questions based on analytical methods listed in the facility's waste analysis plan. The checklists were used during the audits to ensure a thorough audit and were completed by PRC. When negative responses were indicated on the checklist, detailed comments were provided concerning the issue. Comments from completed checklists are incorporated into this report and are grouped as major and minor concerns. The completed checklist for Detrex, including comments, is presented in the Attachment to this report.

The purpose of this document is to summarize the findings of the laboratory audit, including an evaluation of the facility's waste analysis plan and a complete evaluation of the laboratory quality control (QC) program. The following sections summarize laboratory audit activities, provide a laboratory overview, and present major and minor concerns, other comments, and recommendations. References are provided in the final section of this report.

2.0 SUMMARY OF ACTIVITIES

During April and May 1995, PRC conducted a document review of Detrex's current waste analysis plan, with special emphasis on laboratory procedures and QC. From the analytical procedures listed in the waste analysis plan, PRC developed a facility-specific laboratory audit checklist.

On May 17, 1995, PRC representatives Ms. Candace Friday and Ms. Nancy McDonald joined Ms. Jeanette Noechel and Ms. Jan Sealock of MDNR to perform the laboratory audit at Detrex. The audit began with a general briefing with the Detrex operations manager, Mr. Ronald Hritzkowin, and an assistant manager, Ms. Becky Thomas. The laboratory staff consisted of a chemist, Mr. Andres Garcia. Because Mr. Garcia was not available on the day of the audit, several issues could not be adequately addressed. On December 14, 1995, EPA issued a formal request for additional information regarding uncompleted items on the checklist. On January 23, 1996, Detrex submitted the requested information and supporting documentation (Detrex 1996). Information provided in the response was adequate and has been incorporated into the completed checklist. Mr. Hritzkowin and his staff were very cooperative during the entire audit. Audit activities included the following:

- Conducting personal interviews with the operations manager and assistant manager
- Tracking waste sample documentation from prequalification through acceptance
- Reviewing sampling procedures for compliance with the waste analysis plan
- Reviewing analytical techniques for method compliance
- Evaluating the laboratory quality assurance/quality control (QA/QC) program

At the end of the day, PRC conducted a debriefing interview with Detrex and MDNR personnel. During the debriefing, PRC indicated that additional information would be requested at a later date because of the absence of the laboratory chemist. Immediately following the laboratory audit, PRC partially completed the facility-specific checklist. After receiving the response from Detrex, PRC completed the remainder of the checklist attached to this report.

3.0 LABORATORY OVERVIEW

Detrex primarily accepts halogenated solvent liquid waste for reclamation. The Detrex laboratory consists of a single analytical system that analyzes both inbound shipments of wastes as well as outbound treated wastes and recycled solvents. Inbound and outbound shipments of waste can be traced through the analytical system by a unique laboratory identification number. Laboratory personnel include one full-time chemist trained in all fingerprint tests. Instrumentation includes a gas chromatograph (GC) with suitable detectors, pH meter, and Pensky-Martens flashpoint apparatus. In addition, an assortment of general laboratory equipment, such as a hot plates, pH indicator paper, hydrometers, balances, and glassware were present and in working order. The average throughput of samples in the laboratory is about 10 to 20 samples per day.

4.0 MAJOR CONCERNS

This section summarizes comments from the attached checklist that are considered major deficiencies. A deficiency is considered major when one or more of the following issues apply to the activity, procedure, method, or documentation being reviewed:

- Inconsistent with requirements of 40 CFR 264/265 Subparts B and E regarding the following:
 - Current waste analysis plan
 - Recordkeeping requirements
- Insufficient QC as required by Chapter One, Quality Control, of SW-846 (EPA 1994)
- Major modifications to analytical methods cited in the waste analysis plan

Several major deficiencies were noted in the laboratory. The following sections discuss concerns regarding the above-mentioned issues.

4.1

40 CFR 264/265 SUBPARTS B and E

Because Detrex is a permitted facility, the requirements of 40 CFR 264 Subparts B and E apply. The following sections discuss major concerns regarding general waste analysis and the waste analysis plan, as well as recordkeeping deficiencies.

4.1.1

General Waste Analysis and Waste Analysis Plan

Before preparing the facility-specific audit checklist, PRC reviewed Detrex's current waste analysis plan, revised in September 1991. Detrex has included as an attachment to the waste analysis plan a laboratory quality assurance project plan (QAPP), dated July 1991 (Detrex 1991b). The QAPP details QC measures used at the facility laboratory. This section discusses concerns noted in the waste analysis plan and the QAPP. The following deficiencies and inaccuracies were noted:

- The organizational chart of the facility (see Figure 3.1 of the QAPP) shows a laboratory manager, laboratory staff, quality assurance (QA) officer, and sample custodian as the complete laboratory personnel. During the audit, the entire laboratory staff consisted of one chemist who performed all the duties. The current situation is not consistent with the QAPP and does not allow for independent review of any analytical results by a supervisor or QA officer.
- Section 4.2 of the QAPP states that duplicate samples, reagent blanks, and matrix spikes will be analyzed at a frequency of 1 percent or 1 per day, whichever is more frequent. However, during the audit duplicates, matrix spikes, and reagent blanks were not routinely analyzed. The current QC practices are inconsistent with the QAPP and are not sufficient to meet the QC requirements of SW-846 (EPA 1994).
- Section 9.0 of the QAPP states that analytical data reduction and validation is performed under the direction of the laboratory QA officer. As stated above, there is no peer review process in the laboratory system.
- Section C-1b of the waste analysis plan states that based on the analytical testing, "approval is given to sign the manifest and accept the wastes into the container or tank storage areas." During the audit, containers of waste were accepted into the container storage area before analysis and without a signed manifest. The accompanying MDNR inspector, Ms. Noechel, also took note of this practice.

4.1.2 Recordkeeping

Subpart E of CFR 40 265 requires keeping all personnel training records and all records and results of waste analysis. Two major recordkeeping deficiencies were noted during the laboratory audit. In the absence of the laboratory chemist, the managers of the facility and auditors were not able to reconstruct complete analytical sequences for any of the fingerprint and solvent content analyses. According to SW-846 (EPA 1994), records should be kept so that all analyses performed can be reconstructed, including instrument calibrations, calibrations checks, and all associated QC and sample analyses.

In addition, from the records reviewed during the audit, managers of the facility and auditors were not able to determine which analytical methods were used to generate the pH and flammability results. Again, the records being kept were not sufficient to document the analytical procedure used to produce the results.

4.2 QUALITY CONTROL

Because the facility is regulated under RCRA, and because the waste analysis plan and QAPP state that the QA/QC procedures from the SW-846 (EPA 1994) are followed, the facility should perform the minimum QC as required by Chapter One, Quality Control, of SW-846 (EPA 1994) as well as method-specific QC. The following deficiencies noted during the laboratory audit are considered major deficiencies:

- Detrex does not perform duplicate analyses or collect duplicate samples from the same waste stream unless there is a discrepancy between the sample results and the expected results. Duplicate samples should be collected and analyzed at a frequency of 1 per 20 samples to determine sampling precision, analytical precision, and shipment homogeneity.
- Detrex does not perform routine matrix spikes or use surrogate compounds for solvent analysis as required by Methods 8010 and 8015. Because of the nature of the samples, the potential for matrix interferences is great and should be monitored by matrix spike and surrogate recoveries.

- Detrex analyzes a calibration check for solvent content after every 30 samples. As required by Methods 8010 and 8015, the calibration check should be analyzed after every 10 samples.
- For the determination of ignitability, no calibration fluid was available at the time of the audit to check the apparatus. However, Detrex stated in its response that a suitable calibration fluid has been purchased.
- Detrex stated that data received from independent laboratories (for example, Research Technical Institute and Clayton Laboratories) is not validated. However, Detrex also indicated that electronic data received from Clayton Laboratories did not match raw data. This information suggests that a system of data review and validation should be implemented for all data.

4.3 MAJOR ANALYTICAL METHOD MODIFICATIONS

Most analytical methods cited in the waste analysis plan required some degree of modification because of the nature of the samples (for example, spent solvents that are 100 percent organic, as opposed to soils and waters). Modifications are generally considered major when reagents differ, techniques differ, or procedural steps of the method are omitted or substituted. Minor differences are discussed in Section 5.0 of this report. Basically, all modifications to the cited methods should be detailed in the waste analysis plan and listed in laboratory logbooks.

The rule (40 CFR 264 Subpart B) states that test methods used to test for parameters should be addressed in the waste analysis plan. The following analytical method reviewed during the audit was so significantly modified from the methods given in the waste analysis plan that the method cited no longer represents the actual procedures being performed on the waste:

- At the time of the audit, the Detrex laboratory performed flammability testing using a lighted match; however, the waste analysis plan cites ASTM Method D4982, which uses a burner. In its response to the U.S. EPA's information request, Detrex included a new standard operating procedure (SOP) to determine flammability using a propane burner. If this technique has been incorporated into the routine analytical method for flammability since the audit, no further action is required.

5.0 MINOR CONCERNS

This section summarizes comments from the attached checklist that are considered minor deficiencies. A deficiency is considered minor when it is not categorically a major deficiency, and one or more of the following issues apply to the activity, procedure, method, or documentation being reviewed:

- Poor housekeeping
- Slight modifications to methods such as sample size and standard concentrations that do not affect data quality
- Recommended secondary QC requirements are missing
- Outdated methods or equipment

The following minor deficiencies were noted during the laboratory audit.

- Reagent chemicals were not dated with the date received or date opened. To ensure that the chemicals are used on a first-in, first-out basis, all reagent and standard chemicals should be dated. Standard solutions are of particular concern because their quality may deteriorate rapidly.
- Because QC checks, such as matrix spikes and duplicates, were not routinely analyzed by the laboratory, control charts establishing QC limits were not being generated.
- No SOP for pH was available during the audit. As such, there was confusion as to which method of pH determination was being used (for example, indicator paper or pH meter). Detrex's response indicated that the pH meter is inoperable and that pH is determined by indicator paper.
- During the audit, it was apparent that flammability screening was performed by holding a lighted match over the vapors of the sample. However, in its response, Detrex included an SOP, dated January 1996, that indicated a torch was used to determine flammability. The torch method is preferred and should be implemented.

6.0 OTHER COMMENTS

This section summarizes other comments concerning the laboratory that are not categorically major or minor concerns. The following comments do not require any response but provide further characterization of laboratory operations:

- Detrex lists several analyses in the waste analysis plan that it does not perform in-house (for example, tests for reactivity, cyanide, and sulfide). The facility should indicate which methods are actually performed on site, and which are contracted to commercial laboratories.
- In addition to the lack of QC review of analytical results, having only one chemist capable of performing analyses may cause the operation to be slowed or halted if he is not available to perform the required tests. It may be appropriate to train another employee with suitable educational background to assist in the analyses when the chemist is not available. Detrex should document the training and keep the records for future audits.

7.0 RECOMMENDATIONS

PRC recommends that, at a minimum, the laboratory should be required to address all major and minor concerns in writing to MDNR. In addition, PRC recommends that all major concerns should be rectified within a reasonable time frame, and documented proof of the rectification should be submitted to MDNR.

8.0 REFERENCES

- Detrex Chemical Industries, Inc. 1991a. Revised Waste Analysis Plan. September.
- Detrex Chemical Industries, Inc. 1991b. Quality Assurance Project Plan. July.
- Detrex Chemical Industries, Inc. 1996. Letter Response to U. S. Environmental Protection Agency (EPA). Request for Information. Robert Currie, General Counsel. January 23.
- U. S. EPA. 1988. RCRA Laboratory Audit Inspection Guidance Document. Office of Waste Programs Enforcement, RCRA Enforcement Division. OSWER Directive 9950.4 September.
- EPA. 1994. Test Methods for Evaluating Solid Waste, Volumes IA-IC: Laboratory Manual, Physical/Chemical Methods; and Volume II: Field Manual, Physical/Chemical Methods, SW-846. Third Edition. Office of Solid Waste and Emergency Response. Washington DC. September.

ATTACHMENT

RCRA LABORATORY COMPLIANCE EVALUATION INSPECTION CHECKLIST

(44 Sheets)

From: DENNIS WESOLOWSKI
To: R5WST.R5RCRA.SUTKER-SHARI, R5WST.R5RCRA.DEBUS-ALLE...
Date: Tuesday, July 12, 1994 1:10 pm
Subject: Encotec for Detrex

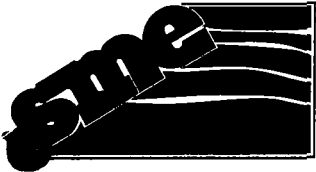
This is a final follow-up to the lab audit request for Encotec.

I spoke with Richard Kuehn of the lab about the proposed 0.4 ppb vinyl chloride detection limit(DL). He said they were unaware of that DL. They had bid on using the CLP SOW which calls for 10 ppb. If the low DL is requested then he will need to re-bid the project.

The bottom line is that we think the lab can do all of the other analyses as indicated in your request. They have done method 524.2 for drinking waters (a clean matrix) down to a DL of 0.5 ppb.

If this low DL is not important and 10 ppb will suffice, then the lab is capable and CASS does not need to be consulted further. If the 0.4 ppb DL will be sought, then you may consider requesting us to review the lab's method detection limit study for that low DL.

At this point I will consider this issue closed. Please contact me at 6-1970 if there are questions or if there are further developments.



soil and materials engineers, inc.

43980 Plymouth Oaks Blvd. Plymouth, MI 48170-2584 (313) 454-9900 FAX (313) 454-0629

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Chuck A. Gemayel, PE
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John C. Zarzecki, CWI

July 11, 1994

Mr. Karl E. Bremer, Chief
United States Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3590

RE: Quality Assurance Project Plan (QAPjP)
Detrex Corporation
12886 Eaton Avenue
MID 091 605 972
SME Project No. PE-21229

RECEIVED

OFFICE OF RCRA
WASTE MANAGEMENT DIVISION
EPA REGION 5

Dear Mr. Bremer:

This letter was written in response to the U.S. EPA's request for additional information with regard to the above referenced site. Mrs. Shari Sutker of the U.S. EPA requested that the cover page of Section 4 (QAPjP) be signed by the appropriate staff of Soil and Materials Engineers, Inc. (SME).

SME has completed the requested task and has included the signatory page as an enclosure with this letter.

If you have any questions or comments on this submittal, you may contact us at (313) 454-9900.

Very truly yours,

SOIL AND MATERIALS ENGINEERS, INC.

Robert J. Nowakowski, CPG
Project Consultant

Enclosure: Cover letter of QAPjP

cc: Rhonda Blayer, MDNR
Bill Moore, Detrex Corporation
Ron Swan, Detrex Corporation

SECTION 4

QUALITY ASSURANCE PROJECT PLAN (QAPJP)

**DETREX - EATON AVENUE FACILITY
DETROIT, MICHIGAN
MID 091 605 972**

U.S. EPA Region V Permit Writer

Shawn Suttel

U.S. EPA Regional Quality Assurance Manager, William A. Debus per MOA
Monitoring and Quality Assurance Branch (MQAB) dated 3/31/94 between
WMD and the OR-Reg-S, and Adm.
Record Log #

SME Project Manager (Robert J. Nowakowski)

Robert J. Nowakowski

SME Quality Assurance Manager (Laura S. Badalamenti)

Laura S. Badalamenti

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5

DATE: 6/14/94

SUBJECT: Laboratory Audit Request

FROM: Richard Traub, Section Chief
RCRA Permitting/Enforcement, HRP-8J

TO: Charles T. Elly, Director
Central Regional Laboratory, SL-10C

Under a RCRA Facility Investigation (RFI), samples will be sent to the following laboratories. The RCRA Corrective Action Plan requires evaluation of laboratories proposed for analyzing RFI samples. Please notify us within 5 days from receipt of this memorandum whether a lab audit and/or performance evaluation is required, and, if this is the case, when will be scheduled.

Facility Name: Dexco State: MI

RCRA Project Coordinator: Shari Suter Phone: 886-6151 Mail Code: HRP-8J

Section Chief: Richard Traub Phone: 886-6136

Laboratory A: Encotech Address: 3985 Research Park Drive, Ann Arbor, MI
Contact: Jim Kuehn Phone: 313-761-1389
Parameters/Groups: See Attached Methods: Contract laboratory Program
list

Laboratory B: Address: _____
Contact: _____ Phone: _____
Parameters/Groups: _____ Methods: _____

TO BE COMPLETED BY CRL: CRL Date In _____ CRL Date Out _____

Laboratory Selected	Evaluation Required? Yes/No	Site Visit Required? Yes/No	Date Assigned for Site Visit	Projected Date for Audit Report
Lab A				
Lab B				

Please return copy of signed memo to A. Debus (RCRA Permitting) or M. DeRosa (RCRA Enforcement) and assigned RCRA Project Coordinator at mail code, HRP-8J.
CRL COMMENTS:

Signature: _____

cc: Dennis Wesolowski, SL-10C
Allen Debus, HRP-8J
Mike DeRosa, HRP-8J

Parameters, Detection Limits, AND Michigan's Act 307
 Type A + B Cleanup levels (ppb)
FOR DETREX CORPORATION

Target Compound	TDL (part per billion)	(ppb) Type B Cleanup levels	(ppb) Type A Cleanup lev.
methylene chloride	10	60	
1,1-dichloroethane	10	14,000	
1,2-dichloroethane	10	8	
1,1,2,2,-tetrachloroethane	10	4	
1,2-dichloroethene (total)	10	8	
1,1,1-trichloroethane	10	4,000	
1,1,2-trichloroethane	10	12	
tetrachloroethene	10	18,000	
toluene	10	14,000	
ethylbenzene	10	6,000	
xlenes (total)	30	120	
chloroform	10	4	
vinyl chloride	10	60	
trichloroethene~	10		

Target method detection limit
for compounds

FOR SOILS ONLY.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

MAY 1 2 1995

HRE-8J

To Whom It
May Concern

Re: Laboratory Audit
Detrex Chemical Industries, Inc.
EPA ID No. MID 091 605 972

Dear Sir or Madam:

The purpose of this letter is to introduce Ms. Candace Friday and Ms. Nancy McDonald as contractors to the United States Environmental Protection Agency (U.S. EPA) under contract number 68-W4-0007, work assignment number R05037. Pursuant to the authority of Section 3007 of the Resource Conservation and Recovery Act, as amended (RCRA) (copy enclosed), 42 U.S.C. §6927, these individuals have been duly designated by the United States Environmental Protection Agency to participate in a RCRA compliance evaluation inspection lead by Michigan Department of Natural Resources, Livonia District Office staff and to request information regarding laboratory practices and hazardous waste.

Your facility is identified in U.S. EPA files as a permitted treatment and storage facility. The purpose of this inspection is to evaluate your compliance with waste analysis plan requirements of your permit and applicable regulations for hazardous waste.

You may, if you desire, assert a business confidentiality claim covering part or all of the information requested, in the manner described by 40 CFR 2.203(b). You should read the above-cited regulations carefully before asserting a business confidentiality claim, since certain categories of information are not properly the subject of such a claim. Information covered by such a claim will be disclosed by U.S. EPA only to the extent, and by means of the procedures, set forth by 40 CFR Part 2, Subpart B. If no such claim accompanies the information when it is received by U.S. EPA, it may be made available to the public by U.S. EPA without further notice to you. Ms. Friday and Ms. McDonald are contractually obligated to comply with applicable confidential business information requirements.



Thank you for your cooperation in this laboratory audit conducted as part of a RCRA compliance evaluation inspection. If you have any questions regarding either the authority to conduct this inspection or the Federal regulations applying to waste analysis plans, please contact Ms. Sue Rodenbeck Brauer, the U.S. EPA Work Assignment Manager, at (312) 353-6134.

Sincerely yours,



Michael DeRosa, Acting Chief
Technical Enforcement Section #2
RCRA Enforcement Branch

Enclosure

cc: Ms. Jeanette Noechel, MDNR WMD Livonia D.O., w/ enc.

An Act

To provide technical and financial assistance for the development of management plans and facilities for the recovery of energy and other resources from discarded materials and for the safe disposal of discarded materials, and to regulate the management of hazardous waste.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled,

SHORT TITLE

SECTION 1. This Act may be cited as the "Resource Conservation and Recovery Act of 1976".

Resource
Conservation and
Recovery Act of
1976.

42 USC 6901
note.

AMENDMENT OF SOLID WASTE DISPOSAL ACT

SEC. 2. The Solid Waste Disposal Act (42 U.S.C. 3251 and following) is amended to read as follows:

"TITLE II—SOLID WASTE DISPOSAL

"Subtitle A—General Provisions

"SHORT TITLE AND TABLE OF CONTENTS

"SEC. 1001. This title (hereinafter in this title referred to as 'this Act'), together with the following table of contents, may be cited as the 'Solid Waste Disposal Act': 42 USC 6901 note.

"Subtitle A—General Provisions

- "Sec. 1001. Short title and table of contents.
- "Sec. 1002. Congressional findings.
- "Sec. 1003. Objectives.
- "Sec. 1004. Definitions.
- "Sec. 1005. Governmental cooperation.
- "Sec. 1006. Application of Act and integration with other Acts.
- "Sec. 1007. Financial disclosure.
- "Sec. 1008. Solid waste management information and guidelines.

"Subtitle B—Office of Solid Waste; Authorities of the Administrator

- "Sec. 2001. Office of Solid Waste and interagency coordinating committee.
- "Sec. 2002. Authorities of Administrator.
- "Sec. 2003. Resource recovery and conservation panels.
- "Sec. 2004. Grants for discarded tire disposal.
- "Sec. 2005. Labeling of certain oil.
- "Sec. 2006. Annual report.
- "Sec. 2007. General authorization.

"Subtitle C—Hazardous Waste Management

- "Sec. 3001. Identification and listing of hazardous waste.
- "Sec. 3002. Standards applicable to generators of hazardous waste.
- "Sec. 3003. Standards applicable to transporters of hazardous waste.
- "Sec. 3004. Standards applicable to owners and operators of hazardous waste treatment, storage, and disposal facilities.
- "Sec. 3005. Permits for treatment, storage, or disposal of hazardous waste.
- "Sec. 3006. Authorized State hazardous waste programs.
- "Sec. 3007. Inspections.

such State and to issue and enforce permits for the storage, treatment, or disposal of hazardous waste unless, within ninety days following submission of the application the Administrator notifies such State that such program may not be authorized and, within ninety days following such notice and after opportunity for public hearing, he finds that (1) such State program is not equivalent to the Federal program under this subtitle, (2) such program is not consistent with the Federal or State programs applicable in other States, or (3) such program does not provide adequate enforcement of compliance with the requirements of this subtitle.

"(c) **INTERIM AUTHORIZATION.**—Any State which has in existence a hazardous waste program pursuant to State law before the date ninety days after the date of promulgation of regulations under sections 3002, 3003, 3004, and 3005, may submit to the Administrator evidence of such existing program and may request a temporary authorization to carry out such program under this subtitle. The Administrator shall, if the evidence submitted shows the existing State program to be substantially equivalent to the Federal program under this subtitle, grant an interim authorization to the State to carry out such program in lieu of the Federal program pursuant to this subtitle for a twenty-four month period beginning on the date six months after the date of promulgation of regulations under sections 3002 through 3005.

"(d) **EFFECTIVE DATE PERMIT.**—Any action taken by a State under a hazardous waste program authorized under this section shall have the same force and effect as action taken by the Administrator under this subtitle.

"(e) **WITHDRAWAL OF AUTHORIZATION.**—Whenever the Administrator determines after public hearing that a State is not administering and enforcing a program authorized under this section in accordance with requirements of this section, he shall so notify the State and, if appropriate corrective action is not taken within a reasonable time, not to exceed ninety days, the Administrator shall withdraw authorization of such program and establish a Federal program pursuant to this subtitle. The Administrator shall not withdraw authorization of any such program unless he shall first have notified the State, and made public, in writing, the reasons for such withdrawal.

"INSPECTIONS

"Sec. 3007. (a) **ACCESS ENTRY.**—For purposes of developing or assisting in the development of any regulation or enforcing the provisions of this title, any person who generates, stores, treats, transports, disposes of, or otherwise handles or has handled hazardous wastes shall, upon request of any officer, employee or representative of the Environmental Protection Agency, duly designated by the Administrator, or upon request of any duly designated officer, employee or representative of a State having an authorized hazardous waste program, furnish information relating to such wastes and permit such person at all reasonable times to have access to, and to call all records relating to such wastes. For the purposes of developing or assisting in the development of any regulation or enforcing the provisions of this title, such officers, employees or representatives are authorized—

"(1) to enter at reasonable times any establishment or other place where hazardous wastes are or have been generated, stored, treated, disposed of, or transported from;

"(2) to inspect and obtain samples from any person of any such wastes and samples of any containers or labeling for such wastes. Each such inspection shall be commenced and completed with reasonable promptness. If the officer, employee or representative obtains any samples, prior to leaving the premises, he shall give to the owner, operator, or agent in charge a receipt describing the sample obtained and if requested a portion of each such sample equal in volume or weight to the portion retained. If any analysis is made of such samples, a copy of the results of such analysis shall be furnished promptly to the owner, operator, or agent in charge.

"(b) **AVAILABILITY TO PUBLIC.**—Any records, reports, or information obtained from any person under this section shall be available to the public, except that upon a showing satisfactory to the Administrator (or the State, as the case may be) by any person that records, reports, or information, or particular part thereof, to which the Administrator (or the State, as the case may be) or any officer, employee or representative thereof has access under this section if made public, would divulge information entitled to protection under section 1905 of title 18 of the United States Code, such information or particular portion thereof shall be considered confidential in accordance with the purposes of that section, except that such record, report, document, or information may be disclosed to other officers, employees, or authorized representatives of the United States concerned with carrying out this Act, or when relevant in any proceeding under this Act.

"(2) Any person not subject to the provisions of section 1905 of title 18 of the United States Code who knowingly and willfully divulges or discloses any information entitled to protection under this subsection shall, upon conviction, be subject to a fine of not more than \$5,000 or to imprisonment not to exceed one year, or both.

"(3) In submitting data under this Act, a person required to provide such data may—

"(A) designate the data which such person believes is entitled to protection under this subsection, and

"(B) submit such designated data separately from other data submitted under this Act.

A designation under this paragraph shall be made in writing and in such manner as the Administrator may prescribe.

"(4) Notwithstanding any limitation contained in this section or any other provision of law, all information reported to, or otherwise obtained by, the Administrator (or any representative of the Administrator) under this Act shall be made available, upon written request of any duly authorized committee of the Congress, to such committee (including records, reports, or information obtained by representatives of the Environmental Protection Agency).

"FEDERAL ENFORCEMENT

"Sec. 3008. (a) **COMPLIANCE ORDERS.**—(1) Except as provided in paragraph (2), whenever on the basis of any information the Administrator determines that any person is in violation of any requirement of this subtitle, the Administrator may issue an order requiring compliance immediately or within a specified time period or the Administrator may commence a civil action in the United States district court in the district in which the violation occurred for appropriate relief, including a temporary or permanent injunction.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5

DATE: JAN 18 1994

SUBJECT: Guidance Concerning Four QAPP Approval Options

FROM: Karl Bremer, Chief
RCRA Permitting Branch

Joe Boyle, Chief
RCRA Enforcement Branch

TO: All RPB and REB Section Chiefs

As many of you are now aware, on December 20, 1993, Norm Niedergang signed a memorandum which grants the Office of RCRA more flexibility concerning the review and approval of QAPPs than was previously the case. For background on circumstances leading to the issuance of this memorandum and the details of the four approval options that were outlined, you are referred to the contents of the December 20, 1993 memorandum. Although any of four options may now be employed, it may not be readily apparent to many of you when to utilize them. Also, because the QAPP review process remains a lengthy one, despite the improvements which are being implemented, together with the Regional goal of obtaining data that is documented to be of a known and reliable quality, a few concepts pertaining to the central theme of targeting project objectives shall be outlined. This guidance is intended to help RCRA Project Officers involved with corrective action scenarios to plan projects effectively from the start with respect to setting pertinent objectives, thereby ensuring that the projects do not become mired in QAPP-related difficulties later on.

A year ago, there were two QAPP approval strategies available to OR. One of these was the standard procedure we have all become familiar with through experience and training. A second option that was available to staff in the RCRA Permitting Branch was referred to as the Bremer Option, as defined in Karl Bremer's memorandum of July 29, 1992. Now, the number of possible options has doubled to four. Since Option 2 is the "Environmental Science Division (ESD) Approval" process already in place through our Memorandum of Agreement between the Waste Management and Environmental Sciences Divisions, emphasis need only be placed on the other 3 options.

Options 1, 3 and 4 require some further definition beyond that provided in Norm Niedergang's December 20, 1993 memorandum to William Sanders. Option 1 is rather limited in scope, pertaining only to situations applicable to either Interim Measures or "Site Stabilization", as formally defined in U.S. EPA guidance. Only cases which meet these descriptions, administratively speaking, should be approved under Option 1.

Option 3 refers to situations where the RCRA QAPP Coordinator may be empowered to recommend approval of a QAPP. This does imply, however, that review of the QAPP will take place in ESD. Only after its initial review has occurred will it be possible for the RCRA QAPP Coordinator to make determinations as to whether ESD's comments have been adequately addressed. If it appears that this has been the case, then the RCRA QAPP Coordinator can recommend that the

Regional Quality Assurance Manager (RQAM) sign the title page of the QAPP. (See page 4 of Norm Niedergang's December 20, 1993 memorandum for further details.) It is possible, however, that facilities may not have addressed the ESD concerns satisfactorily, and that the RCRA QAPP Coordinator will be unable to recommend approval to ESD. ESD must concur with the RCRA QAPP Coordinator's recommendation, and the RQAM must sign the QAPP's title page based on this recommendation, before an Option 3, "RCRA QAPP Coordinator Approval" has resulted.

Datex
An Option 4, "RCRA Project Coordinator Approval" will allow "program approval" of a QAPP that has been reviewed previously by ESD. "Program approval" may be interpreted to mean Branch level approval of a QAPP. (See the December 20, 1993 memorandum, mentioned previously, for more details concerning which QAPPs would qualify for this option.) As is the case with Option 3, there must have been some level of review performed by ESD before the Office of RCRA may elect to approve a QAPP, either conditionally or unconditionally. One service provided by ESD which may fulfill this requirement is participation in Quality Assurance Section (QAS)-RCRA "scoping" meetings, combined with the RCRA requested laboratory audit. The RCRA QAPP Coordinator may request that the Central Regional Laboratory (CRL) perform an evaluation of the proposed laboratory facility(ies) via the "Laboratory Audit Request" form. The proper use of this form shall be described below. Essentially, if the QAPP has been scoped and reviewed through the RCRA program, and project objectives have been clearly established, then, following the CRL's evaluation of all proposed laboratories, it may be possible for the OR to approve the QAPP.

The Laboratory Audit Request form may be used only after project objectives have been clearly established by the RCRA project officer and the "team" of U.S. EPA players involved in the project. The "team" will include the roster of individuals assigned to the project, collectively including Office of RCRA personnel, State representatives, and individuals from both the Central Regional Laboratory and the Quality Assurance Section. Although it is now recommended to conduct "scoping" and "pre-QAPP" meetings before the QAPP has been drafted by the Permittee, it may not be possible for the team to agree on a well defined set of project objectives until after the QAPP has been prepared and reviewed at least once. Therefore, it is impermissible for anyone to request an audit or evaluation of the proposed laboratory(ies) until after the QAPP has been received and reviewed thoroughly by those parties, designated by the RCRA project officer as being involved in the approval process. (The Laboratory Audit Request form is intended to be used whenever audits are needed, so its use is clearly not restricted only to the Option 4 approval strategy.)

One reason why laboratory audits should not be requested until after the QAPP has reached an advanced stage of preparation is that the CRL will require a very specific document for review, including all standard operating procedures (SOPs) that will be utilized during the course of your investigations. If a preliminary document is submitted to CRL for review, any fundamental changes to sampling methods, matrices, or analytical procedures may invalidate its review. It is vital that we do not waste resources in requesting CRL's assistance until after a definitive project has been planned through the QAPP review process.

It may be the case that some QAPPs destined for Option 4 approval may be approvable following the CRL's recommendation of a laboratory for your respective projects. Such approval may result from the CRL's review without requested assistance from the Quality Assurance Section. However, it is evident that Option 4 approvals cannot proceed to the point where the CRL's assistance can be requested until a great deal of planning and review has been conducted beforehand. Guidance on "how to proceed" is outlined below.

PLANNING AND REVIEW STAGES

The most commonly encountered difficulty with RCRA QAPPs lies in the general area of setting pertinent project objectives. This is one area where ESD usually provides many significant comments back to the OR's Project Officers. The problem is compounded for us because it is very difficult to establish a list of action levels that the data will be eventually compared to. There are a variety of approaches which have been taken by OR staff, some founded on a bit of ingenuity, but all arrived at through onerous soul-searching and anxiety.

From experience, frustration on all sides often results when the facility representatives offer certain laboratory "packages" purported as being capable of meeting our "objectives", only to find that the ESD doesn't want to entertain any discussion of analytical methodologies until after the target objectives (e.g., parameters and method detection limits that are needed) have been completely established. Unfortunately, it is usually the case that the RCRA Project Officers, on behalf of the assigned "team", have not yet concluded what these objectives are until after the QAPP has been reviewed several times by QAS.

This situation hampers our program not because of staff-related inadequacies or ill-conceived programmatic intentions, but mainly because of a general lack of guidance and discourse in this area. This memorandum is not intended to resolve this thorny problem. This may be one of the primary reasons it takes so long to approve RCRA QAPPs. Some of you have suggested that a special workgroup dedicated to the goal of establishing procedures for how RCRA project objectives can be set and implemented should be formed. It may be the case that the Region will have to develop and follow its own guidance for RCRA, and that a means of routinely evaluating how objectives should be set would be a focus of this special committee.

Until the Region develops any guidance on this matter, there are a number of materials which may help you in establishing objectives which flesh out the general corrective action requirements. For instance, the May 23, 1993 Region 5 Model QAPP instruction for "QAPP Element 3, Project Description" contains some advice intended for the facility to follow. Another U.S. EPA document, "Getting Ready Scoping the RI/FS", November 1989, is intended for use in the Superfund program, but may be of some help to the RCRA program as well.

In the meantime, it is recommended that all OR staff consider the following items and procedures when the impending deadline for a new QAPP approaches:

1. Hold an internal scoping meeting about 1 week prior to the scheduled meeting date for the "pre-QAPP" meeting. Participants should include all individuals who bear responsibility for reviewing and approving the QAPP including representatives from QAS and CRL. Discussions should include familiarization of the team to the facility and the important background issues (i.e., difficult matrices, recalcitrant facility, "low" prioritization, etc.)
2. Prior to the "pre-QAPP" meeting, to the maximum extent possible, attempt to familiarize yourselves with the body of historical data already available, which may have some bearing on some RFI or phase I RFI decisions which may be made concerning project objectives. Consider whether you are comfortable with the quality of this data. How much of this data will you accept? Can this information be utilized in lieu of repeated testing? Ask questions, and do try to write down your conclusions in memo format addressed to your administrative records.
3. At the "pre-QAPP" meeting, attempt to learn what the facility thinks the project objectives are. Then, discussion can center on whether or not these objectives are reasonable. Once your team agrees on objectives, focus discussion on intended target parameters and method detection limits that are pertinent. Matters concerning analytical strategies and appropriate numbers of samples are secondary concerns which can not really be decided until after everybody on the team understands and agrees with the intended project objectives.
4. After each meeting with the facility, do record your observations and outline what was discussed in a memorandum addressed to your administrative records. Always distribute copies of your meeting summary memoranda to the other members of your team. Remember, you are the "captain" of the team, and it should be your responsibility to keep everyone informed. Try to avoid meetings with facility representatives unless an agenda has been prepared by the facility in advance of the meeting.
5. When it finally appears that resolution over what the pertinent project objectives will be and what the intended data usages are, prepare a memorandum which will be distributed to all members of your team. It may not be possible for resolution to be reached on this matter until after the QAPP has been reviewed several times, but keen foresight and planning may eliminate the problem of "iteration".
6. Consult with your RCRA QAPP Coordinator for further insights on how the task of setting proper objectives may relate to your projects.

This memorandum will not address all your questions concerning this tricky but fundamental area. However, it is intended that this shall provide internal guidance on "how to proceed" with the review and approval of QAPPs now that the OR has made four basic options available to staff. If you have any further questions concerning this memorandum, please contact Mr. Allen A. Debus at 886-6186 or Mike DeRosa at 353-7992. Please share this memorandum with your respective staff.

cc: George Schupp, QAS
 Dennis Wesolowski, CRL
 Norm Niedergang, OR

CONCURRENCE REQUESTED FROM REB			
SEC/BR SECRTRY			
OTHER STAFF	REB STAFF	REB SECTION CHIEF	REB BRANCH CHIEF
	<i>M. P.</i> 1/12/94	<i>JD</i> 1/13/94	<i>CEP</i> 1/13/94

CONCURRENCE REQUESTED FROM RPB			
SC/BR SECRTRY			
OTHER STAFF	RPB STAFF	RPB SECTION CHIEF	RPB BRANCH CHIEF
<i>1/11/94</i> <i>Proctor</i> <i>RB</i>	<i>AD</i> 1/11/94	<i>CBP</i> 1/11/94	<i>CEP</i> 1/8/94

DETREX CORPORATION

P.O. Box 5111, Southfield, MI 48086-5111
May 13, 1993

FAX: (313) 358-5803

Regional Administrator
U.S. EPA Region V
77 W. Jackson Street
Chicago, IL 60604

RE: Seibert Oxidermo
Spill Report No. 163853
March 23, 1993

Dear Sir,

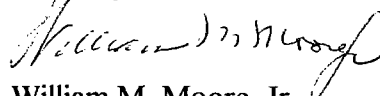
As indicated in our written response of April 7, 1993 concerning the above release, Detrex Corporation parent corporation of Seibert Oxidermo, Inc. is providing the results of the analytical data received from samples taken at the discharge area. Samples taken were sent to have corrosivity, ignitability, and reactivity run along with TCLP metals and organic compounds. Results of soil samples show non-detect on all analysis.

From the accompanying results, Detrex and Seibert believe the discharge area was cleaned up in accordance with applicable federal and state regulations, and that no further remediation is needed.

As noted in our initial report the cause of the discharge from the containment area was because of improper construction of the containment dike. This containment area has been placed "out of service" until the contractor has an opportunity to correct the dike.

If additional information is needed, please do not hesitate to contact me.

Sincerely,



William M. Moore, Jr.
Corporate Manager
Environmental Compliance
RCRA Sections

enclosure
cc: Michigan DNR
file

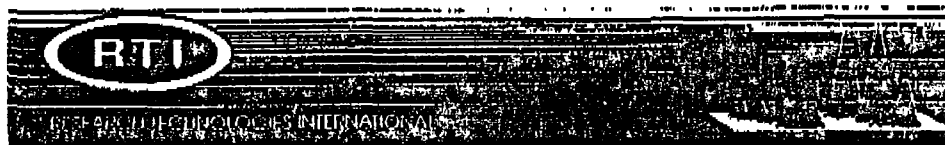
0: WMD
CC: WATER (LO)
WESTLAKE (LO)
RA RF (LO)

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PROTECTION AGENCY
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TELEPHONE:
(313) 358-5800

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REGIONAL OFFICE

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RTI LABS


RTI

RESEARCH TECHNOLOGIES INTERNATIONAL

LABORATORY ANALYSIS REPORT

Submitted by : Bob Anderson

Lab. No. : 93-2827A

Client : Earth Services
Birmingham

Report Date : 04/12/93

Sample ID :

Seibert-Oxidermo
1 soil sample

Object :

Analyze for full TCLP (minus herbs and pests), and RCI

Results and Discussion :**SAMPLE IDENTIFICATION**

1 - Stockpiled Soil

SAMPLE MATRIX

Soil

Parameter *****	Result *****	Regulatory Limit *****
Corrosivity (pH units)	8.8	2 - 12.5
Ignitability (Flash point, deg. F)	>200	> 200
Reactivity		
Total Available Cyanide (ppm)	<1.0	250
Total Available Sulfide (ppm)	<1.0	500

TCLP Metals

Parameter *****	Result (ppm) *****	Regulatory Limit (ppm) *****
Arsenic	<0.200	5
Barium	0.380	100
Cadmium	<0.020	1
Chromium	<0.020	5
Mercury	<0.0002	0.2
Lead	<0.050	5
Selenium	<0.150	1
Silver	<0.020	5
Copper	<0.010	100
Zinc	<0.010	500

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LABORATORY ANALYSIS REPORT
Lab. No. : 93-2827A

Page : 2

SAMPLE ID :
Saibert-Oxidermo
1 soil sample

TCLP ORGANIC PORTION

Parameter	MDL (ppm)	LOD-S (ppm)	Result (ppm)	Regulatory Limit (ppm)
VOLATILE ORGANIC COMPOUNDS				
Benzene-----	0.005	0.005	ND	0.5
Chloroform-----	0.005	0.005	ND	6.0
Carbon tetrachloride-----	0.005	0.005	ND	0.5
Chlorobenzene-----	0.005	0.005	ND	100.0
1,4-Dichlorobenzene-----	0.010	0.010	ND	7.5
1,2-Dichloroethane-----	0.005	0.005	ND	0.5
1,1-Dichloroethylene-----	0.005	0.005	ND	0.7
Methyl ethyl ketone-----	0.100	0.100	ND	200.0
Trichloroethylene-----	0.005	0.005	ND	0.5
Tetrachloroethylene-----	0.005	0.005	ND	0.7
Vinyl Chloride-----	0.010	0.010	ND	0.2
SEMI VOLATILE ORGANIC COMPOUNDS				
Total Cresols-----	0.01	0.01	ND	200.0
O-Cresol-----	0.01	0.01	ND	200.0
m-Cresol-----	0.01	0.01	ND	200.0
p-Cresol-----	0.01	0.01	ND	200.0
2,4-Dinitrotoluene-----	0.01	0.01	ND	0.13
Nitrobenzene-----	0.01	0.01	ND	2.0
Hexachlorobenzene-----	0.01	0.01	ND	0.13
Hexachloro-1,3-butadiene-----	0.01	0.01	ND	0.5
Hexachloroethane-----	0.01	0.01	ND	0.13
Pyridine-----	0.01	0.01	ND	5.0
Pentachlorophenol-----	0.05	0.05	ND	100.0
2,4,5-Trichlorophenol-----	0.05	0.05	ND	400.0
2,4,6-Trichlorophenol-----	0.05	0.05	ND	2.0

LABORATORY ANALYSIS REPORT
Lab. No. : 93-2827A

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SAMPLE ID :
Seibert-Oxidation
1 soil sample

QA/QC DATA**1. SURROGATE & RECOVERY**

	Result(%)	Limits(%)
VOLATILE COMPOUNDS		
1,2 Dichloroethane-d4-----	91	76- 114
Toluene-d8-----	96	88- 110
4-Bromofluorobenzene-----	98	86- 113
SEMI-VOLATILE COMPOUNDS		
2-Fluorophenol-----	92	21- 100
Phenol-d5-----	63	10- 94
Nitrobenzene-d5-----	101	35- 114
2-Fluorobiphenyl-----	125**	43- 116
2,4,6-tribromophenol-----	61	10- 123
Terphenyl-d14-----	121	33- 141

2. METHOD BLANKS (ORGANICS):

FILE ID	FRACTION	COMPOUND	RESULT (ppm)
HPB1630	VOA	Methy ethyl Ketone	ND
		Acetone	ND
		Freon	ND
		All Other Compounds	ND
HPA4069	SEMI-VOL	All compounds	ND

3. METHOD BLANKS (METALS):

	INSTRUMENT BLANK (ppm)	METHOD BLANK (ppm)	MDL (ppm)	METHOD
As	ND	ND	0.200	6010
Ba	ND	ND	0.005	6010
Cd	ND	ND	0.020	6010
Cr	ND	ND	0.020	6010
Hg	ND	ND	0.0002	7040
Pb	ND	ND	0.050	6010
Se	ND	ND	0.150	6010
Ag	ND	ND	0.020	6010
Cu	ND	ND	0.010	6010
Zn	ND	ND	0.010	6010

LABORATORY ANALYSIS REPORT
Lab. No. : 93-2827A

Page : 4

SAMPLE ID :
Seibert-Oxidermo
1 soil sample

4. MATRIX SPIKES, DUPLICATES, & CHECK STANDARDS (METALS): (ppm)

	SPIKE ADDED	SAMPLE RESULT	MS RESULT	% REC.	MSD RESULT	% REC.	RPD	INST. STD (%)	METHOD STD (%)
As	4.00	0.073	3.92	96	4.22	104	7.3	0.0	101
Ba	4.00	0.384	4.30	98	4.42	101	2.8	102	102
Cd	4.00	0.0	3.34	84	3.59	90	7.2	100	93
Cr	4.00	0.002	3.84	96	4.03	101	4.8	98	98
Hg	4.00	0.0	3.43	NA	NA	NA	NA	101	100
Pb	4.00	0.005	3.44	86	3.65	91	5.9	97	104
Se	4.00	0.045	4.03	90	4.52	112	11.5	103	100
Ag	4.00	0.0	3.74	94	4.00	100	6.7	103	101
Cu	4.00	0.0	3.99	100	4.52	108	7.9	101	93
Zn	4.00	0.024	3.97	99	4.09	102	3.5	96	97

SAMPLE PROCESSING DATA

	METALS	VOLATILE COMPOUNDS	SEMI-VOLATILE COMPOUNDS
Date Extracted/Digested:--	04/06/93	NA	04/01/93
Extracted/Digested by:---	CY	NA	KR
Date Analyzed:-----	04/05/93	04/06/93	04/01/93
Analyzed by:-----	CY	RE	RE
File ID:-----	NA	HPH1631	HFA4070
Batch #:-----	040593	040693	040193
Dilution:-----	NA	NONE	NONE

Notes:

* Also present in blank, results have been blank corrected.
 ** QC criteria not met.
 *** No surrogates recovered due to matrix interference and number of dilutions needed to analyze the sample.
 LOD-S Limit of Detection for the reported sample.
 MDL Method Detection Limit.
 NA Not Applicable or Not Analyzed.
 ND Not Detected or less than MDL.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

copy 2

DATE: NOV 02 1992

SUBJECT: QAPJP Review for Dextrex Corporation
MID 091 605 972

FROM: Charles B. Slaustas, Chief
Minnesota/Wisconsin Permitting Section

Charles B. Slaustas

TO: Richard Traub, Chief
Michigan Permitting Section

Ms. Shari Kolak of your staff requested Allen Debus of my staff to review a quality assurance project plan (QAPJP) for the facility referenced above. The QAPJP was received on October 16, 1992.

The attached General Comments are intended for Ms. Kolak's information, but with some restructuring could be submitted to the facility in a notice of deficiency letter along with the attached Specific Comments.

It is recommended that three guidance documents be sent to Detrex to facilitate their revision of the QAPJP. Although their response should focus on the enclosed comments, it may be instructional for Detrex representatives to read the "Region 5 Model RCRA QAPJP," dated, May 1991, and the "Contents Requirements for RCRA RFI QAPJPs," dated October 1990. Detrex' contractors may wish to substitute "boiler-plate" language from the model QAPJP into sections of the Detrex QAPJP concerning issues such as chain of custody, data quality objectives, and quality assurance objectives for measurement data. A third guidance manual "Specifications and Guidance for obtaining Contaminant-Free Sample Containers", dated April 1990, has been referred to in comment 18. Mr. Debus has provided Ms. Kolak with copies of these documents.

If it is necessary to make changes, Mr. Debus has placed the comments on "f:\user\share\kolak\". If you have any further questions or comments concerning this review, Mr. Debus may be contacted at 6-6186. Please forward this information to Ms. Kolak.

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GENERAL COMMENTS REGARDING THE QAPJP for DETREX CORPORATION OF DETROIT, MI

Currently, use of Test Methods for Evaluating Solid Waste, SW-846, 3rd edition, is preferred for all RCRA Facility Investigatory work. Detrex has proposed use of the CLP for Organic Analysis, August, 1991, with two exceptions (see page 30 of the QAPJP). CLP is normally utilized in the Superfund program. However, under exceptional circumstances the CLP could be allowed for use in RCRA, particularly if the assigned permit writer accepts the facility's rationale for using CLP. If, as may be the case here, the facility's laboratory contractual agreement is structured on the use of CLP capability, rather than SW-846, for example, this may form a basis for why CLP should be used. One reason for why the Detrex QAPJP was so brief compared to others usually submitted to address QA issues in the RCRA program is because CLP rather than SW-846 protocols have been proposed. Consequently, Detrex' contractor, Testing Engineers & Consultants, Inc., has simply incorporated the CLP Statement of Work (SOW) by reference. The CLP SOW can be regarded as a 'cookbook' approach to analytical testing, whereas SW-846 is definitely not a 'cookbook'. Therefore, if SW-846 methods were required for this project, the contents of this QAPJP would be considerably expanded. Because the CLP SOW is tailored for the low/medium contamination range, higher range contamination may require use of SW-846 methods. Since there is no clear indication in the QAPJP of how high the noted contamination is, it may be difficult at present to make any such determination. However, for the purposes of this review, it has been assumed that use of the CLP SOW can be approved. Consequently, if Detrex intends to use the CLP SOW, then references to proposed methods, and laboratory control limits for these constituents must be presented in the QAPJP. A statement that CLP SOW will be followed without deviation should also be incorporated. According to a representative of U.S. EPA's QAS, tentatively identified compounds (TICs) should be reported (see page 30 of the QAPJP).

Secondly, the target parameters selected for the RFI activity may require further consideration. Only twelve constituents have been proposed (see Table 1, page 14 of the QAPJP). However, there is little rationale presented supporting why it is this list alone that bears relevance to the Detrex project. Previous studies have indicated evidence of contamination, but Detrex' contractor claims that this information cannot be validated. Also, it is never indicated what constituents were analyzed during these previous studies, although it is mentioned that 1,1,1 trichloromethane was detected. On page 8 of the QAPJP, there is indication that Detrex handles a variety of materials, including some which may contain metals. The historical use of the facility prior to 1950 was never discussed. Test method capability was not indicated, but I suspect that the CLP SOW method that they plan on using would allow them to quantify more compounds than just the 12 proposed ones, if present. By agreeing to report data only for a subset of constituents which can be analyzed by a particular method, there is risk that others which are present and which would be quantitatively measured anyway would not be reported to the U.S. EPA. The facility should list, rather than incorporate by reference, the intended contract required detection limits for each target constituent, (see page 14 of the QAPJP). It is unclear whether the tests they will be performing will be conducted on a TCLP extract, in accordance with 40 CFR 261.24, to determine characteristics of extracted soil, or whether they will be analyzing solutions that have been extracted in a more aggressive manner (e.g. see section 7.4.3 of

method 8240, SW-846, 3rd edition). The outcome of this decision will ultimately depend on the overall project objectives. However, if the permit writer would like to explore this issue (TCLP) further, then elaboration to comment 12 may be required. Detrex should be encouraged to address and or clarify these matters before submitting a QAPjP revision.

SPECIFIC COMMENTS CONCERNING THE QAPjP for DETREX CORPORATION OF DETROIT, MI

1. Although previously generated data could not be validated, some further indication of historical data results must be presented in order to aid reviewers in determining whether the proposed target analyze list is suitable. This information should include lists of all constituents which were analyzed and the detection limits obtained for these analyses. There should be indication of the sampling locations and depths for each set of analyses.
2. It should be indicated in the QAPjP whether a subcontractor will be utilized for analytical services. The facility name and location(s) which shall receive samples for analysis must be presented so that the U.S. EPA can schedule a laboratory audit.
3. There are several objectives mentioned in section 1.0 of the QAPjP. However, if possible detailed project objectives should also be determined and added to the QAPjP. For instance, are there other specific purposes for generating data, besides determining the vertical and horizontal extent of contamination at Detrex? Does a need exist for eventually comparing data to any known action level criteria or health based standards (State or Federal)? Are there any data quality objectives or detection limit criteria which must be met in order to create the proposed contour figure indicating contaminant concentration levels.
4. In the Introduction to the QAPjP, it should be indicated who owned the site prior to 1950, and what the facility was operated for. (See page 8 of the QAPjP.)
5. Where is it planned to establish soil "background" levels? A rationale of how background constituent levels will be determined must be presented in the QAPjP.
6. As mentioned in comment 1, there should be full presentation of any other parameters, besides select volatile such as 1,1,1 trichloromethane, that were analyzed previously.
7. A greater rationale should be presented in the QAPjP as to why only 12 constituents shall be analyzed during the RFI. This is needed because there are informational gaps in the QAPjP concerning facility history prior to 1950 and results of previous sampling. Also, based on Detrex' nature of business, as explained on page 8 of the QAPjP, metals containing materials are handled on site. At a minimum, there should be discussion included in the QAPjP explaining why other classes of compounds such as metals and semivolatiles should be excluded from consideration.

8. For each target constituent, the intended contract required detection limit should be presented, not simply referenced.
9. In reference to section 2.6 of the QAPjP, evidence substantiating the statement that no contamination exists below 15.0 feet should be presented?
10. Only 12 volatile constituents have been selected for analysis. However, other volatile which can be analyzed using the method proposed for analyzing the 12 should be reported if detected. Unless there is strong rationale presented as to why data for other volatile constituents for which the method has been validated is not needed, then results for these constituents should also be reported. (e.g. See SW-846, method SW-846, Table 6, in the case of a volatile scan using gas chromatography/mass spectrometry. Although it is understood that Detrex plans to use the Contract Laboratory Program Statement of Work (CLP SOW) for this project, the method actually selected should be very similar to analogous SW-846 methods.) This rationale should indicate whether or not it is possible that Detrex may receive materials for recycling which may possibly be contaminated with other low level volatile organic constituents.
11. In Figure 2 of the QAPjP, it is evident that there are no plans to sample underneath existing structures, even though on pages 2 and 3 of the Pre-Investigation Evaluation of Corrective Measures Technologies (PECMT) report, it is indicated that such data may at some time be needed. This apparent discrepancy should be discussed. (See page 2 of the PECMT report and page 16 of the QAPjP.)
12. Is it intended to analyze soil samples only for TCLP-leachable (without bias correction) volatile constituents, or will a more aggressive extraction procedure be performed?
13. Referring to page 21 of the QAPjP, specificity is needed when discussing "relatively high levels of data precision, accuracy, and completeness". The laboratory must include its recent, actual control limits for proposed target constituents, in lieu of presenting data quality objectives acceptance criteria in terms of accuracy and precision and completeness. Tabular format is preferred. For RCRA project work, greater than 90% completeness is expected.
14. Referring to section 5.3 of the QAPjP, there should be no field blank for soils.
15. A proposed frequency for matrix spikes and matrix spike duplicates (MS/MSD) should be presented. MS/MSD samples are required for organic analysis. Samples designated for MS/MSD analyses will be collected with extra volumes, at a frequency of one per group of 20 or fewer investigative samples. Triple the normal sample volumes will be collected for volatile organic constituents. (Twice the normal sample volumes will be collected for extractable organics, pesticides and PCBs.)

16. What will the frequency of trip blank collection be?
17. For volatile organic constituents, the holding time for samples is 14 days from the period of time from collection to that of analysis for samples preserved with acid.
18. In reference to page 25 of the QAPjP, bottle blank analyses should be conducted. Criteria which all containers must meet to be considered acceptable should be added to the QAPjP.
19. Procedures for use of sample tags (in addition to sample labels) should be specified.
20. Although use of analytical methods selected from SW-846, Test Methods for Evaluating Solid Waste, 3rd edition, is preferred for RCRA work, under special circumstances, use of the CLP SOW may be accepted. However, if methods are selected from the CLP SOW, then references to method section numbers should be indicated in the QAPjP.
21. In section 8.0 of the QAPjP it is stated that tentatively identified compounds (TICs) shall be not be reported. However, for RCRA purposes, TICs must be identified and reported.
22. A CLP-like deliverable data package must be made available to the U.S. EPA upon request. Generally, 100% data validation is preferred for RCRA work.
23. Preventive Maintenance, as presented on page 33 of the QAPjP, must address field equipment.
24. In section 13.0, page 33 of the QAPjP, equations to assess data precision, accuracy, and completeness must be provided.



NATURAL RESOURCES COMMISSION
MARLENE J. FLUHARTY
GORDON E. GUYER
D. STEWART MYERS
RAYMOND POUPORE

JOHN ENGLER, Governor

DEPARTMENT OF NATURAL RESOURCES

STEVENS T. MASON BUILDING
P.O. BOX 30028
LANSING, MI 48909

DELBERT RECTOR, Director

August 13, 1991

Mr. Bill Moore
Corporate Engineering & Risk Management
Detrex Corporation
P.O. Box 5111
Southfield, Michigan 48086-5111

Dear Mr. Moore:

SUBJECT: Background Soil and Groundwater Data
Collection Program
Detrex Corporation, MID 091 605 972

The Waste Management Division (WMD) has completed a review of the Background Soil and Groundwater Data Collection Program that was submitted on August 8, 1991. The program was submitted in response to Deficiency E.1.b of the WMD's August 2, 1991, Notice of Deficiency for Detrex Corporation's (Detrex) revised Act 64 operating license application.

The WMD hereby approves the Background Soil and Groundwater Data Collection Program subject to the following modifications:

1. Soil samples will be collected and analyzed from the top six inches of the native sand, and from the sand and clay at the interface between the sand and clay units.
2. Decontamination of the augers, cutting bits, and other drilling equipment, and all sampling equipment must be conducted in an area constructed and designed to collect the decontamination solutions and prevent run-off and run-on. The decontamination solutions may not be discharged to the surface away from the borehole locations. All of the decontamination solutions must be containerized, characterized, and subsequently managed in accordance with all federal and state regulations.

It is the WMD's understanding that the data collection program is scheduled to begin by August 16, 1991. Given the holding times of the samples and the time that Detrex has requested to review the data, the data must be submitted to the WMD by September 20, 1991.

#B

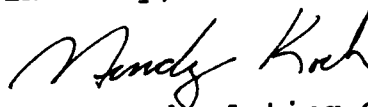
Mr. Bill Moore

-2-

August 13, 1991

If you have any questions, please contact Ms. Ronda L. Hall, Waste Management Division, Department of Natural Resources, P.O. Box 30241, Lansing, Michigan 48909, or at telephone number 517-373-9548.

Sincerely,




Mindy Koch, Acting Chief
Waste Management Division
517-373-9523

cc: Mr. Ronald C. Hritzkowin, Detrex Corporation
Ms. Lorraine Kosik, U.S. EPA
✓ Mr. Rich Traub, U.S. EPA
Mr. Steve Buda, DNR
Ms. Ronda L. Hall, DNR
Mr. Donald Mbamah, DNR-Livonia
Operating License File

DETREX INTER-OFFICE CORRESPONDENCE

TO: M.J. Tepatti
CC: File
SUBJECT: Quality Control Coordinator

FROM: 
R.E. Swan
DATE: 10/27/90

As per your request, a more detailed description of the activities/responsibilities of the newly created position of Quality Control Supervisor is as follows:

POSITION: Quality Control Coordinator

JOB DESCRIPTION: Position is responsible for ensuring that the facility operates within the Hazardous Waste Operational Parameters specified in the Facility Part B Application, City of Detroit Dept. of Water & Sewage, Wayne County Department of Health Air Pollution Control Division, and Corporate established product standards.

Primary Responsibilities:

- 1) Receive samples of hazardous waste at the facility, log in samples, complete waste sample profile sheets for samples, prepare samples for shipment to the appropriate analytical facility, logging in of analytical results, and dissemination of results to Salesmen.
- 2) Ensure that all shipments of non-flammable waste received at the facility are properly Manifested, labeled, marked with accumulation start date, sampled, logged into the Hazardous Waste Operating log, prepared for shipment to appropriate analytical facility or analytical results from generator/transporter are obtained, production control cards are prepared, and bench top fingerprint analysis are preformed.
- 3) Ensure that all shipments of flammable waste received at the facility are properly Manifested, labeled, marked with accumulation start date, labeled with Stream Code approval number, logged into the flammable waste operating log, and coordinate shipments/pick-ups with drivers to allow for optimum use of equipment.
- 4) Coordinate treatment of waste water including supervision of treatment, subsequent sampling and disposal of effluent and filtered solids, shipment of samples and wastes, operation of waste water treatment process equipment, and the use and stocking of treatment chemicals.

M.J. Tepatti
Page Two

- 5) Coordinate Air Monitoring Program including monitoring/sampling of influent and effluent from carbon absorption unit, logging in of results, operation of carbon absorption unit, stocking of materials for use in unit, and coordination of monitoring program with Wayne County Department of Health, Air Pollution Control Division.

Secondary Responsibilities:

- 1) Completion of Production Control cards for invoicing/crediting of customers.
- 2) Conducting titration for non-amine acceptance of reclaimed solvents, calculating amount of stabilizer concentrate is required in reclaimed solvent, and stocking of chemicals required for N.A.A. tests and test kits.
- 3) Logging in production figures and shipment information, including completed manifests and certificates of destruction from TSDF's into hazardous and flammable waste log books. Also responsible for assimilating data required and for putting together certificates of destruction and sending same to customers.

Regulatory Responsibilities:

In addition to the abovementioned, the following regulatory responsibilities shall be undertaken by the position:

- 1) Maintain a file for Import and Export shipments of hazardous wastes for the current year. This data shall be compiled at the end of the year and used to complete the Import/Export Annual Report. The report shall be filed with the U.S.E.P.A. and MI D.N.R. and other parties requiring it not later than the end of the first calendar week of the proceeding year.
- 2) Obtain samples of the effluent from the facility in a timely fashion each quarter of the year. The samples shall be forwarded to an independent laboratory for City of Detroit Water Analysis. Once the analytical results are received, the data shall be utilized to create the six month compliance report for the facility. The report shall be forwarded to the facility manager for timely review and submission to the City of Detroit.

Salary and performance reviews shall be given annually in January. The reviews shall use as a basis of performance the efficiency of completion of the abovementioned tasks as pertaining to the position.

DETREX CORPORATION

CHEMICALS DIVISION • P. O. BOX 1398 • ASHTABULA, OHIO 44004
TELEPHONE 216 997-6131

RECEIVED
APR 17 1989

April 12, 1989

OFFICE OF RCRA
Waste Management Division
U.S. EPA, REGION V

Mr. Dave Petroski
USEPA
Region V
230 South Dearborn Street
Chicago, Illinois 60604

Dear Mr. Petroski:

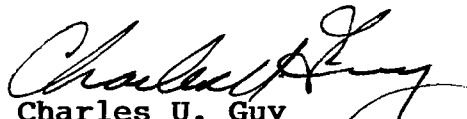
As per your telephone request of March 31, 1989, I have enclosed analytical results for samples of rain water from our transfer station area sump. Samples 89-010 and 012 are the rain water samples.

Please note that the analyses run were City of Detroit parameters, since we intend to discharge this rain water to the city sewer and not to the gravel yard if acceptable to the City.

If you require additional information or have any further questions, please advise.

Sincerely,

DETREX CORPORATION



Charles U. Guy
Manager Environmental Compliance
Corporate Risk Management Dept.

bz

#18

CAL

Canton Analytical Laboratory, Inc.
ENVIRONMENTAL ANALYSIS

March 2, 1989

GOLD SHIELD SOLVENTS DIVISION

Detrex Corporation

12886 Eaton Aven

Detroit, MI 48227

ATTENTION: Ronald E. Swan, Jr.

RE: CAL Report #1364

4 samples delivered 2/13/89

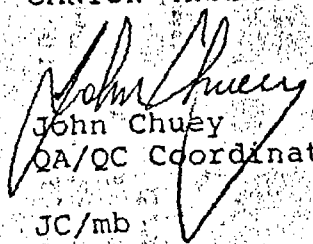
Dear Mr. Swan:

The sample(s) we received from you has/have been analyzed as requested. The results are compiled in the enclosed report.

It is a pleasure to be of assistance to you. Please contact us if you have any questions concerning any aspect of this work.

Sincerely,

CANTON ANALYTICAL LABORATORY, INC.



John Chuey

QA/QC Coordinator

JC/mb

SAMPLES RECEIVED 02/13/89

GOLD SHIELD SOLVENTS

PAGE 1

LAB# 9020538
 LAB# 9020539
 LAB# 9020540
 LAB# 9020541

OUTFALL SAMPLE 89-010
 WASTE WATER SAMPLE 89-011
 OUTFALL SAMPLE 89-012
 SAMPLE #89015

LAB#
 UNITS

G.S. Det.

G.S. Det.

G.S. Det.

Elco Waste Water
 (Untreated)
 9020541
 mg/kg

City of De
 Pretreat
 Stand

yanide, Total
 Total Suspended Solids
 BOD, 5 Day
 Phosphorus, Total
 Silver, Total
 Arsenic, Total
 Cadmium, Total
 Copper, Total
 Lead, Total
 Mercury, Total
 Nickel, Total
 Chromium, Total
 Zinc, Total
 PCB'S BY AROCHLOR

9020538 mg/l	9020539 mg/l	9020540 mg/l	9020541 mg/kg	
< 0.02	< 0.02	< 0.02	< 0.05	2.0
5.0	3.0	15	3100	10,000
14	50	100	21000	10,000
0.14	0.02	4.8	4.5	500
0.10	0.10	0.08	0.02	2.0
< 0.002	< 0.002	< 0.002	2.8	1.0
0.03	0.03	0.04	< 0.01	2.0
0.02	0.03	0.07	0.11	4.5
< 0.05	< 0.05	< 0.05	0.05	1.0
< 0.0005	< 0.0005	< 0.0005	< 0.01	.005
0.06	0.06	0.06	0.04	5.0
0.03	0.03	0.03	0.04	25.0
0.04	0.05	0.22	0.07	15.0

Canton Analytical Laboratory

GOLD SHIELD SOLVENTS

SAMPLES RECEIVED 02/13/89

PAGE 2

LAB# UNITS	9020538 ug/l	9020539 ug/l	9020540 ug/l	9020541 ug/l	City of Det. Pretreatment Standards
PCB-1016	< 1.0	< 1.0	< 1.0	< 10	1.0
PCB-1221	< 1.0	< 1.0	< 1.0	< 10	"
PCB-1232	< 1.0	< 1.0	< 1.0	< 10	"
PCB-1242	< 1.0	< 1.0	< 1.0	< 10	"
PCB-1248	< 1.0	< 1.0	< 1.0	< 10	"
PCB-1254	< 1.0	< 1.0	< 1.0	< 10	"
PCB-1260	< 1.0	< 1.0	< 1.0	< 10	"
PCB-1262	< 1.0	< 1.0	< 1.0	< 10	"
PCB, Total	< 1.0	< 1.0	< 1.0	< 10*	"
Oil and Grease, mg/l	33	< 1.0	72	1900	2000